STOPPING
WATER POLLUTION
AT ITS SOURCE

MISA
Municipal/Industrial Strategy for Abatement

MISA LABORATORIES

QUALITY ASSURANCE & ANALYTICAL METHODS WORKSHOP

PROCEEDINGS
MARCH, 1992

Ontario
Environment
Environnement

PROCEEDINGS/PRESENTATIONS

FROM

MISA ANALYTICAL WORKSHOPS

MARCH 10 & 11, 1992

MARCH 17 & 18, 1992

Laboratory Services Branch
Quality Management Office
125 Resources Road
Rexdale, Ontario
M9W-5L1
(416) 235-6005

April 1, 1992

Dear MISA Workshop Participant,

Many of the participants of the MISA Analytical Workshops held March 10, 11, 17 and 18th have expressed interest in a hard copy of the information presented.

We have produced a proceedings/presentations document for each presentation package order form that the Quality Assurance Office received. Please share this with your colleagues who may have also attended, or other interested parties. As noted in the document introduction, please feel free to copy and use any portions you feel would be useful to your staff.

Cammy Lynn Raposo

MISA Analytical Workshop
Co-ordinator

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SECTION ONE

INTRODUCTION

TOPIC ABSTRACTS

WORKSHOP ASSESSMENT ABSTRACTS

The 1992 MISA Analytical Workshops, hosted by the Environment Ontario, Laboratory Services Branch held March 10 & 11, 17 & 18th, provided the opportunity for laboratory technicians, scientists and managers to present and exchange information related to the analytical support of Ontario's Municipal/Industrial Strategy for Abatement (MISA) Program.

Over the two 2-day workshops, through formal presentations, small workgroups and open discussion, a lot of information and ideas were tabled.

As a direct result of participation and comments from you and your colleagues, Ministry Laboratory staff were able to re-design the second workshop (March 17, 18) to be more effective. This is identified in the assessment survey summary which follows.

As a follow-up the the workshop, and as a means of capturing most of the information presented, this proceedings/presentations document was compiled.

These proceedings provide hard copies of information presented, and in most cases, in the exact format (slides, overheads) used by the authors. This was done intentionally to promote further use and discussion by workshop attendees. Please feel free to copy and use any portions you feel would be beneficial to your colleagues and staff.

On behalf of Environment Ontario Laboratory Services Branch, Management and staff, thank you for attendence and participation.

G. Crawford
Manager, Trace Organics Section & Quality Assurance
Laboratory Services Branch
April 2, 1992

MISA ANALYTICAL WORKSHOPS 1992

MARCH 10 & 11/ 17 & 18

SPEAKER ABSTRACTS

DAY ONE (SESSION ONE)

A) MISA OVERVIEW

Patricia Baulu, MOE/Lab Services Branch
Tel: (416) 323-2770
Fax: (416) 323-2785

A brief historical overview of the MISA Program is given. The status of the MISA program is discussed as well its future direction.

B) CODE OF PRACTICE DOCUMENT

George Crawford, MOE/Lab Services Branch
Tel: (416) 235-5757
Fax: (416) 235-6110

Several Code of Practice documents exist here in Canada and internationally in Europe and should be considered when setting up a laboratory. These documents are briefly reviewed and contacts are provided.

C) NEW DEVELOPMENTS WITH NIMMP

Peter Campbell, MOE/Lab Services Branch
Tel: (416) 235-5872
Fax: (416) 235-6107

A general overview of NIMMP is given. New improvements in the approach taken to carry a procedure through the NIMMP process are discussed.

D) BULLETIN BOARD

Cammy Lynn Raposo & David Boomer, MOE/Lab Services Branch
Tel: (416) 235-6005/5858
Fax: (416) 235-6110/6113

Electronic communication has now become a fast and efficient way of "sharing information." The LSB QM Bulletin Board is introduced by way of discussion and a visual demonstration.

E) LAB QC PRACTICES

David Boomer, MOE/Lab Services Branch
Tel: (416) 235-5858
Fax: (416) 235-6113

Advances in computerized instrumentation has not only increased instrumentation sensitivity but has also improved on analyte selection and QC practices. Two of these systems in place at the LSB are QC-Expert and LQAS.

DAY ONE (SESSION TWO)

F) ROUND ROBINS - MOE LAB PERSPECTIVE

Sylvia Cussion, MOE/Lab Services Branch
Tel: (416) 235-5842
Fax: (416) 235-6110

The Ministry of the Environment Lab Services Branch has been co-ordinating Round Robins since 1988. This presentation will provide a brief overview of the program and some examples of past round robin data.

G) ROUND ROBINS - COMMERCIAL LAB PERSPECTIVE

Judith Szekely, Hamilton-Wentworth Regional Laboratory
Tel: (416) 546-4454
Fax: (416) 545-7177

Brian Fowler, Axys Analytical Laboratory
Tel: (604) 656-0881
Fax: (604) 656-4511

Round robin studies are conducted and particpated in by many laboratories. In different perspectives, presentations are provided by both a Regional laboratory and a commercial laboratory.

H) ROUND ROBINS - WTC PERSPECTIVE

Mohammad Foroutan & Peter Fowlie, Wastewater Technology Centre
Tel: (416) 336-4855
Fax: (416) 336-4765

The Wastewater Technology Centre in Burlington has played a major role in conducting round robins in both water and sediment matrices. The WTC will provide an overview of how they conduct round robin studies and some past examples of round robin data.

I) ANALYTICAL CHEMISTRY UNDER REGULATIONS / JAWG

Dr. Otto Herrmann, Ontario Hydro
Tel: (416) 231-2877
Fax: (416) 231-9679

When regulations are imposed on laboratories, analytical chemistry becomes more challenging. These challenges are discussed in a industry/commercial/government laboratory group called JAWG (Joint Analytical Working Group).

DAY TWO (SESSION THREE)

J) PROTOCOL DOCUMENT

Patricia Baulu, MOE/Lab Services Branch
Tel: (416) 323-2770
Fax: (416) 323-2785

The Protocol for the Sampling/Analysis of Municipal/Industrial Wastewater is introduced. Parts of the document are displayed in the presentation to familiarize the participants with its features.

K) POTENTIAL NON-COMPLIANCES/ENFORCEMENT

Cathy Doehler, MOE/Lab Services Branch
Tel: (416) 235-6055
Fax: (416) 235-6110

Inspections of industry and laboratories are required under the MISA regulation. This presentation focuses on the LSB and their role in inspections and enforcement of the MISA regulation.

L) DETECTION AND LOW-LEVEL COMPLIANCE/ENFORCEMENT

Don King, MOE/Lab Services Branch
Tel: (416) 235-5838
Fax: (416) 235-6110

With the completion of the MISA Monitoring phase, the MISA quality control data is being submitted to the LSB for review. This presentation provides a comprehensive overview of the fundamental concepts of quality control and the interpretation of low-level data.

M) CAEAL UPDATE

Bill Traversy, CAEAL/
Tel: (613) 562-2200
Fax: (613) 562-2203

George Crawford, MOE/Lab Services Branch
Tel: (416) 235-5757
Fax: (416) 235-6110

The Canadian Association for Environmental Analytical Laboratories (CAEAL) is introduced. Information is provided about membership, certification and accreditation.

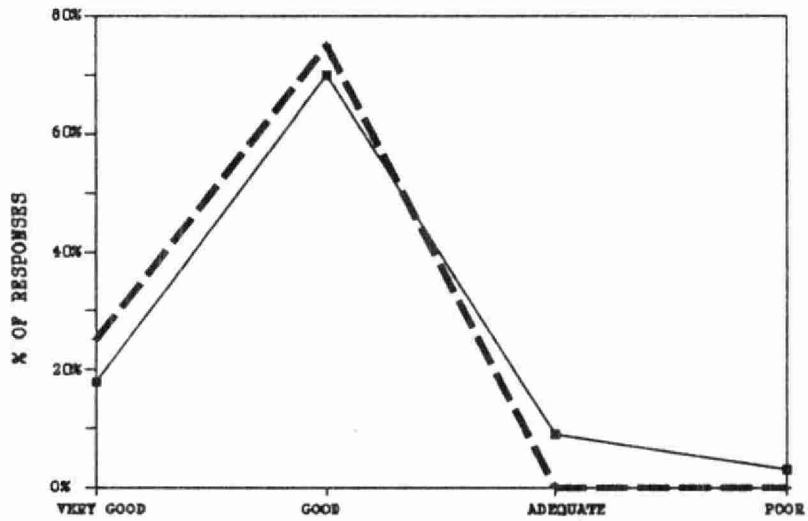
SUMMARY OF PARTICIPANTS ASSESSMENT OF
THE MISA WORKSHOPS 1992

At Session 1 (March 10, 11), there were 73 participants with 35 returning their questionnaires. At Session 2 (March 17, 18), there were 50 participants with only 10 returning questionnaires. Based on those questionnaires received, graphical representations of the summarized results are shown below.

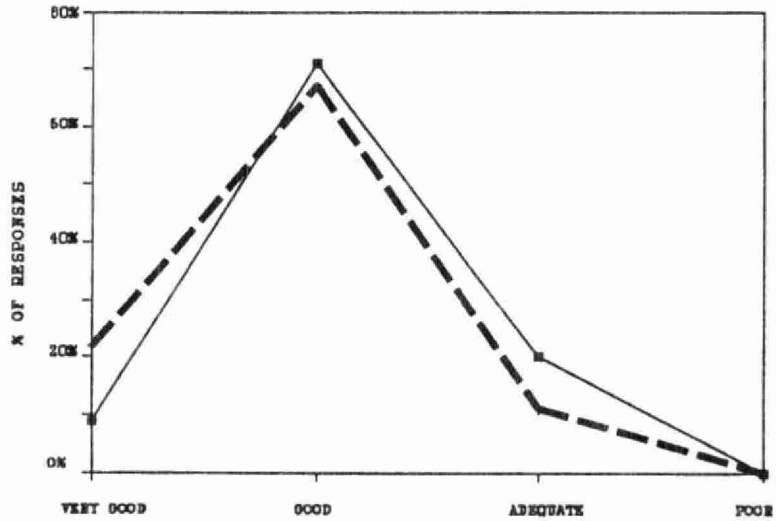
SESSION 1 IS REPRESENTED BY _____

SESSION 2 IS REPRESENTED BY ----------

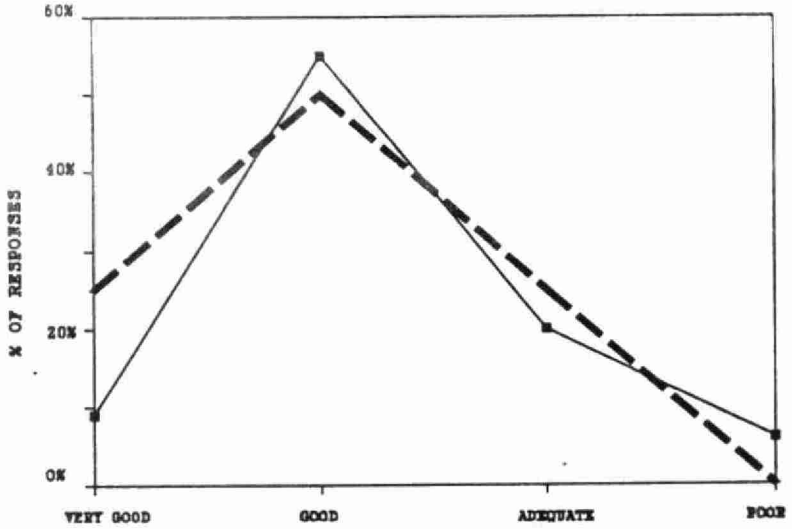
QUALITY OF PRESENTATIONS



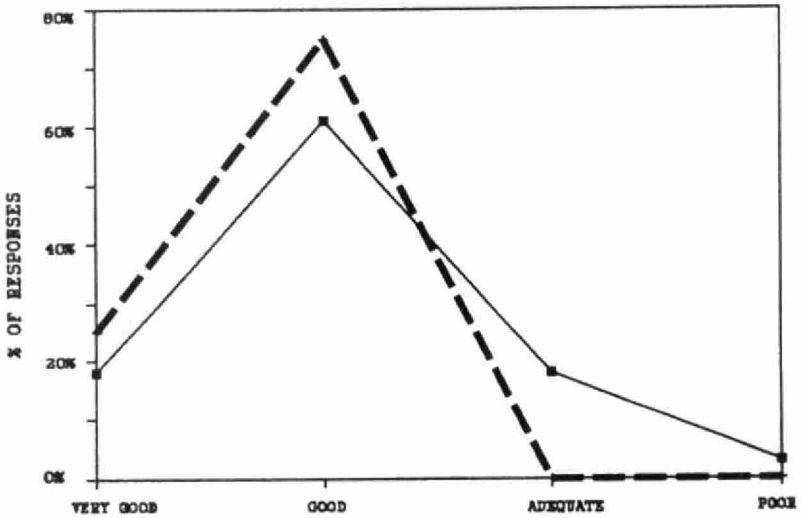
FORMAT OF WORKSHOPS



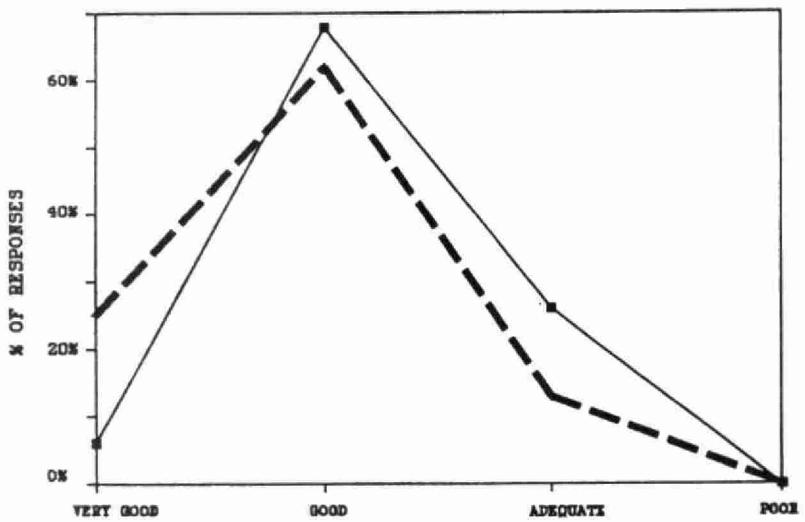
GUANTITY OF INFORMATION



WORKSHOPS



OVERALL ASSESSMENT



SECTION TWO - Day 1 - Session One Presentations

SECTION TWO

DAY 1 - SESSION ONE PRESENTATIONS

CHAIRPERSONS - B. BERG, MOE
C. DOEHLER, MOE

MISA OVERVIEW -	P. BAULU, MOE
CODE OF PRACTICE DOCUMENT -	G. CRAWFORD, MOE
NEW DEVELOPMENTS WITH NIMMP -	P. CAMPBELL, MOE
BULLETIN BOARD -	C. RAPOSO & D.BOOMER, MOE
LAB QC PRACTICES -	D. BOOMER, MOE

OVERVIEW

MUNICIPAL INDUSTRIAL STRATEGY FOR ABATEMENT

MISA

MISA OVERVIEW

Background

OFFICIALLY ANNOUNCED: JUNE 1986 WHITE PAPER

PURPOSE: REGULATE DISCHARGES

OBJECTIVE: VIRTUAL ELIMINATION OF PERSISTENT TOXICS FROM INDUSTRIAL/MUNICIPAL DISCHARGES

APPROACH: DISCHARGERS MONITOR EFFLUENTS LIMITS SET BASED ON DATA & BATEA

JTC's: REGULATIONS DEVELOPED IN CONSULTATION WITH INDUSTRY, MAC, ENVIRONMENT CANADA.

MISA OVERVIEW

Background

INDUSTRIAL SECTORS:

9 SECTORS COVERING 300 DIRECT DISCHARGERS

Petroleum Refining
Organic Chemical Manufacturing
Iron & Steel
Inorganic Chemical
Pulp & Paper
Metal Mining
Metal Casting
Industrial Minerals
Electric Power Generation

MISA OVERVIEW

Background

MUNICIPAL SECTOR:

DIRECT DISCHARGERS: 400 STP's

INDIRECT DISCHARGERS: 12 000 +

22 sectors

MISA OVERVIEW

Current Status

MONITORING: COMPLETED FOR ALL SECTORS

DATA EVALUATION: 4 SECTORS COMPLETE

5 SECTORS IN PROGRESS

CLEAN-UP OF DATABASE FOR EACH PLANT:

Correction of data entry errors
verification of outliers

QA/QC ASSESSMENT:

validate data for use in setting limits

MISA OVERVIEW

Issues Resolution

ISSUES RELATED TO LIMITS SETTING RESOLVED AND PUBLISHED BY MOE IN CONSULTATION WITH INDUSTRY

PURPOSE: SET-UP STANDARDIZED SET OF PROCEDURES AND CRITERIA TO ENSURE CONSISTENCY ACROSS SECTORS

MISA OVERVIEW

Issues Resolution

ISSUES:

BAT and BATEA
Selection of parameters
Monitoring data evaluation
QA/QC
Limits setting and form of limits
Monitoring for assessment
Reporting to the public
Virtual elimination
Flow measurement accuracy
Net loadings
By-passes
Stormwater
Toxicity
Compliance

MISA OVERVIEW

Limits setting

SELECTION OF PARAMETERS:

- compounds selected as candidates for limits
- list derived from computer data base according principles set out in IRC report
- only parameters validated in QA/QC assessment are selected for BAT evaluation
- only parameters for which treatment exists (BAT) are targets for limits
- draft limits are calculated using statistical treatment outlined in IRC report
- draft limits are reviewed/approved within MOE

MISA OVERVIEW

Limits setting

- "FINAL" LIMITS PRESENTED TO JTC FOR CONSULTATION
- LIMITS REGULATION, DEVELOPMENT DOCUMENT & ASSOCIATED PROTOCOLS UNDERGO PUBLIC REVIEW
- LIMITS REGULATION PROMULGATED

CURRENT STATUS:

- DRAFT LIMITS DEVELOPED FOR PETROLEUM & PULP & PAPER SECTORS
- LIMITS UNDER REVIEW WITHIN MOE

MISA OVERVIEW

New Initiatives

ZERO DISCHARGE: ELIMINATION OF PERSISTENT TOXICS FROM EFFLUENTS DISCHARGED TO THE ENVIRONMENT THROUGH BANS & PHASE-OUTS

TOXICS REDUCTION: REDUCTION/SUBSTITUTION OF TOXICS USED AND/OR RELEASED

POLLUTION PREVENTION: ELIMINATE IN-PLANT USE AND PRODUCTION OF TOXICS AS OPPOSED TO END-OF-PIPE TREATMENT

MISA OVERVIEW

Summary

REGULATIONS: EXPECTED FOR PUBLIC REVIEW IN SPRING 1992 FOR PULP & PAPER AND PETROLEUM

DATA REPORTS: PUBLISHED FOR IRON & STEEL AND ORGANIC SECTORS

QA/QC REPORTS: UNDERGOING FINAL REVIEW IN IRON & STEEL, ORGANIC AND MINING SECTORS PRIOR TO FINAL PARAMETER SELECTION/LIMITS

DATA BASE CLEAN UP: NEARING COMPLETION

REGULATIONS: SCHEDULED FOR END OF 1992

ONTARIO MINISTRY OF ENVIRONMENT

LABORATORY SERVICES BRANCH

MISA ANALYTICAL WORKSHOPS

MARCH 10 & 11, 1992

MARCH 17 & 18, 1992

QUALITY MANAGEMENT

**Doing the
right things
right the first time,
on time,
all the time —
and always
to the customer's
satisfaction!™**

CODE OF PRACTICE/ DOCUMENT(S) ISE

- Definition
- Application
- Content
- Sources

CODE OF PRACTICE/ DOCUMENT(S)
ISE

Definition

A Collection of Rules or Canons, Systematically Arranged, that circumscribe the conduct, organization and operation of an analytical laboratory

CODE OF PRACTICE/ DOCUMENT(S) ISE

- Definition
- Application
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- Sources

CODE OF PRACTICE/ DOCUMENT(S)
ISE

APPLICATION

- A COP is essential to establishing and demonstrating the viability of an analytical Laboratory
- All Accreditation and Certification Programs demand an outline of COP information as a mandatory requirement (CAEAL, SCC, CSA, A2LA, IAETL)
- Demonstrated adherence to a COP is one measure of a Laboratory's suitability to provide "MISA Service"

National Accreditation
Program for Testing
Organizations

Directory of Accredited Testing Organizations

CAN-P-1550
April 1988
Fourth Edition

Published by the
Standards Council of Canada
350 Sparks Street, Suite 1200
Ottawa, Ontario, K1P 6N7

INTRODUCTION

This Directory contains a list of accredited testing organizations and the registered detailed scopes of their accredited testing capabilities. Some of these involve the calibration of testing and measuring equipment; such "calibration laboratories" are included in the generic term "testing organizations". Accredited testing organizations meet the criteria of the Standards Council of Canada document *Accreditation Criteria and Requirements for Testing Organizations* (CAN-P-4A).

The accreditation criteria deal with the adequacy of administration, human and physical resources, quality system procedures and freedom from conflict of interest. The criteria are compatible with ISO/IEC Guide 25 *General Requirements for the Technical Competence of Testing Laboratories.*

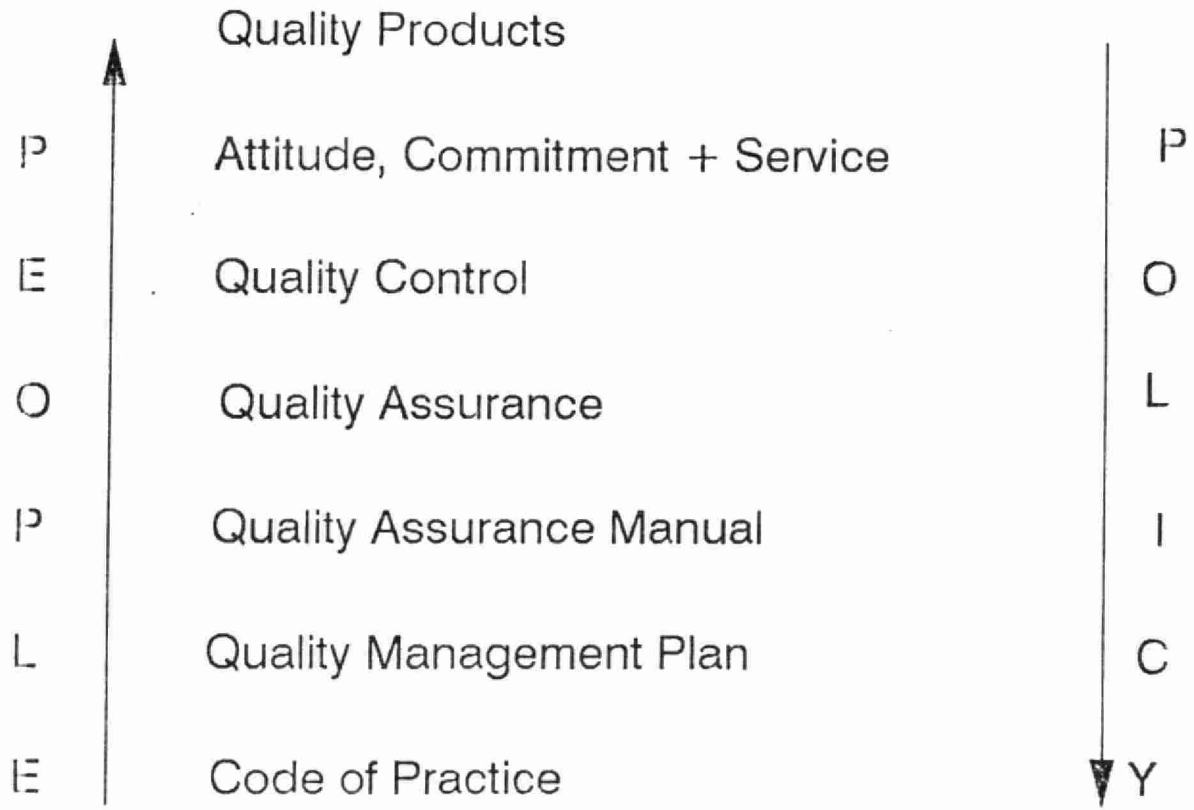
Accreditation is the formal recognition of the capability of an organization to manage and perform specific tests registered with the Council. It does not guarantee individual test results, nor is it a guarantee that test results will be accepted by product certification bodies.

Accreditation does not limit an organization to conducting only those tests for which it is accredited. Most accredited organizations carry out other tests. A client may request that an accredited testing organization indicate in its report whether or not a test is registered.

The role of the Standards Council is to recognize competent testing organizations in Canada, through accreditation, and to provide the relevant information to potential clients. Business matters between an accredited testing organization and its clients are legal transactions strictly between the two parties.

This Directory is primarily intended to promote the services of accredited testing laboratories to users and potential users of testing services. It also serves to publicize the program to organizations which could benefit from

CODE OF PRACTICE/ DOCUMENT(S)
ISE



CODE OF PRACTICE/ DOCUMENT(S)
ISE

- Definition
- Application
- Content
- Sources

INTERNATIONAL STANDARD ISO 9004

INTERNATIONAL STANDARD ISO 9003

INTERNATIONAL STANDARD ISO 9002

INTERNATIONAL STANDARD ISO 9001

INTERNATIONAL STANDARD

ISO 9000

First edition
1987-03-15

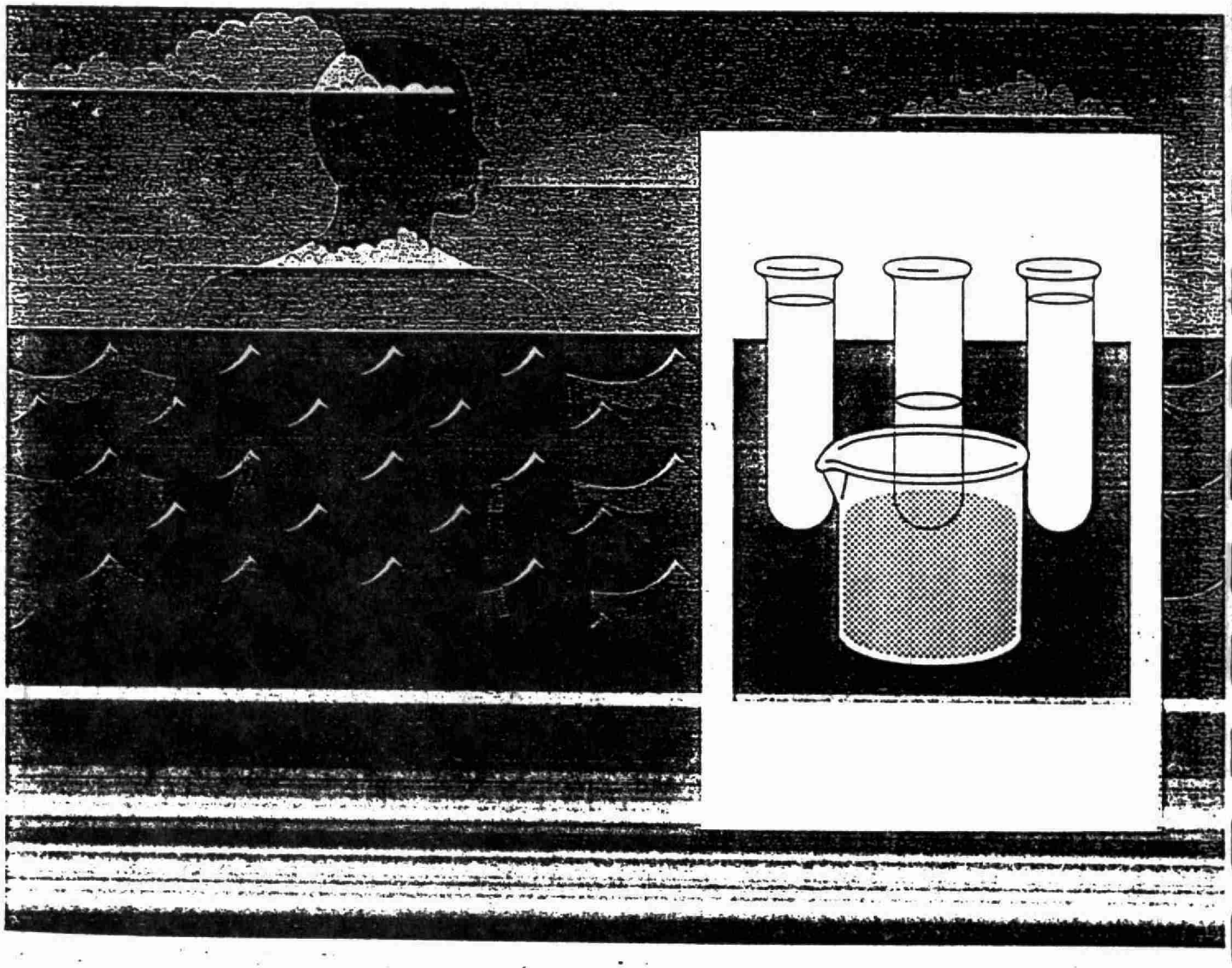
INTERNATIONAL ORGANIZATION FOR STANDARDIZATION
ORGANISATION INTERNATIONALE DE NORMALISATION
МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Quality management and quality assurance standards — Guidelines for selection and use

Normes pour la gestion de la qualité et l'assurance de la qualité — Lignes directrices pour la sélection et l'utilisation

Canadian Standards Association

Z201-M1990
Qualification Code for Ontario for Laboratories Analyzing Industrial Waste



Contents

CROSS-REFERENCE LIST OF QUALITY SYSTEM ELEMENTS

Title	ISO 9001 Q91	ISO 9002 Q92	ISO 9003 Q93	ISO 9004 Q94
Management Responsibility	4.1 o	4.1 x	4.1 *	4
Quality System Principles	4.2 o	4.2 o	4.2 x	5
Quality in Marketing	4.3 o	4.3 o	-	7
Quality in Specification/Design	4.4 o	-	-	8
Quality Documentation/Records	4.5 o	4.4 o	4.3 x	17
Quality in Procurement	4.6 o	4.5 o	-	9
Purchaser Supplier Product	4.7 o	4.6 o	-	-
Material Control/Traceability	4.8 o	4.7 o	4.4 x	11
Quality/Control of Production	4.9 o	4.8 o	-	10
Product Verification	4.10o	4.9 o	4.5 x	12
Control of Measuring/Test Equipment	4.11o	4.10o	4.6 x	13
Control of Verification Status	4.12o	4.11o	4.7 x	11
Nonconformity	4.13o	4.12o	4.8 x	14
Corrective Action	4.14o	4.13o	-	15
Handling/Post-Production Functions	4.15o	4.14o	4.9 x	16
Quality Records	4.16o	4.15o	4.10x	17
Auditing the Quality System	4.17o	4.16x	-	5
Personnel	4.18o	4.17x	4.11*	18
After-Sales Servicing	4.19o	-	-	16
Use of Statistical Methods	4.20o	4.18o	4.12x	20
Economics	-	-	-	6
Product Safety and Liability	-	-	-	19

KEY: o = Full requirement
* = Less stringent than Q92
x = Less stringent than Q91
- = Element not present

Source: ANSI/ASQC Standard Q90-1987

CODE OF PRACTICE

FOR

ENVIRONMENTAL LABORATORIES

SEPTEMBER 1989

Environment
Ontario

CODE OF PRACTICE FOR ENVIRONMENTAL LABORATORIES

Table of Contents

DRAFT

COMPARISON OF REQUIREMENTS IN STANDARDS (*)

DRAFT

ISO Guide 25 General Requirements for Technical Competence of Testing Labs.
EN 45001 General Criteria for the Operation of Testing Labs.
ANSI/ASQC 92 Model for QA in Production and Installation.
EPA 40 CFR Pt 792 TSCA GLP*
EPA 40 CFR Pt 160 FIFRA GLP*
FDA 21 CFR Pt 58 GLP*
ANSI/ASQC Q2 Quality Mgt. & Quality Sys. Elements for Laboratories-Guidelines

The intent of the comparisons is to show that the ANSI/ASQC Standard covers the majority of the requirements in the laboratory standards and adds some additional requirements, which may have to be addressed by the laboratory that is doing manufacturing control, lot acceptance, or support work. The subject is addressed in the paragraphs indicated but it is not intended that the exact wording is the same or that the same level of detail is required. If a single section number addresses basically the same subject and all subheadings, it is listed only once. The initial breakdown was on Guide 25, therefore, the subjects are arranged in the order shown in Guide 25.

If there is a topic which could not be located, there will be "--" under that standard heading.

Since ANSI/ASQC 92 addresses "Production" rather that test laboratory the following was used as a corollary for the laboratory:

Product - Test results and reports
Processes - The test methods and other procedures
Calibration - Standardization
Raw Materials - Reagents, etc.
Quality Assurance system - The system normally used to produce products but not generally found is laboratories as an organized separate activity.
Service - Equals product. The laboratory service (product) is test results, technical competence and Assurance.

* Require a detailed protocol and other biological requirements related to non-clinical studies but the requirements are being applied to testing laboratories that supply identity, purity, physical property, and specification testing for materials being subjected to the non-clinical studies. Only those paragraphs related to "testing" are referenced. Those related to non-clinical studies (animal, biological, etc.testing) are not included. However if "animals" were used, the sections of the GLPs which cover their handling, generally, have the same types of requirements as those listed under reagents, equipment, facilities, and records in the other standards.

DRAFT

DRAFT

(*) Adapted from "Quality Assurance for the Analytical Laboratory",
The Centre for Professional Advancement, East Brunswick, New Jersey, USA.
Jack E. Weiler, Dow Corning Corp., Mich.

Comparison of Standards

Subject	25+	45001	Q92	FDA/EPA#	2	MOE
Scope and field of application	1++	1	1.1/1.2	.1/.10	2	1++
Testing laboratories						4++
Specific location						-
Specific methods						-
Statement of Compliance	--	--	--	--/.12##	--	-
Definitions	2(5)*	2(13)	3(A3)**	.3	4(11)	2(9)/8(8) 9(7)/10(5
Organization		3	4.1.2	Sub B		4
Legally identifiable	3					
Organization chart		5.1			5.3	4.2
Quality system					5.2	3++
Competence		5.1	1.2.1		6.2	-
Freedom		4				-
Extent and limits of responsibility		5.1			5	2++
Technical management		5.1/5.4		.31/.33	14.1.2	3.4
Proprietary and confidential security		5.4.6		.31f		-
Current training	--	5.2	--	--	14.1	4.3
Responsible management	--	5.1	--	--	6.2.1	3
Quality System	4.	5.4.2	4.2	.35	5.2/6.0	2
Quality Policy	--	5.2	4.1.1		5.1	3
One responsible individual	4.1	5.4.2	4.1.2.3		5.3	-
Quality Manual	4.2		4.2	.81	6.3/5.3	3.4
Structure (chart)			4.1.2			
Operational and functional duties			4.1.2.1			3.5.1
Quality Assurance procedures, general					6.2	3.5.2
Quality Assurance procedures, for tests					6.2.5	3.5.3
Proficiency testing, standards, etc.						3.5.5
Corrective action procedures	--	--	4.13		9.4/16/17	-
Complaint procedure, technical		4.3	4.12.1	.31g	12.0	-
Contract review	--	--	4.3			-
Change control(document)	--	--	4.4			8++
Management Quality System Review periodic & documented	4.3	5.4.2	4.1.3		18.2	3++
Quality Loop	--	--	--	--	6.1	-

\+ = The listing was established based on Guide 25 order.
++ = When a single number is shown at the top of a section, it generally covers as subsections and additional numbers indicate that the subject is addressed in other places.
* ( ) = Number of definitions listed.
** A = ANSI/ASQC A3 & ISO8402
\# = Subsection reference only
= --/.12 means not in FDA GMP but in the EPA GMPs

Comparisons of Standards
Page 3

Subject	25	45001	Q92	FDA/EPA#	2	MOE
Staff	5.1	5.	4.1.2.2/	.29	14.0	4.2
Education, training, & technical knowledge	5.1		4.17			4.3
Job descriptions for senior technical	5.2					4++
Adequate supervision	5.3					4.2.3
Suitable staff to cover absence	5.4			.31b		-
Records of qualifications, & training	5.4				14.4	4.3.3
Management	--	--	--	.31	--	4.2
Equipment			4.10	.61	9	7++
Adequate equipment for scope	6.1	5.3.1		.31e & .61		7.2
Proper maintenance of equipment	6.2	5.3.3		.63b		7.2.4
Control of unusable equipment (Tags)	6.3	5.3.1				7.2.3
Use of outside equipment	--	5.3.1				-
Records for each major item:	6.4/6.5/	5.3.3	4.9.1	.63c		-
Name of equipment	6.6					-
Manufacturer's name, type & serial No.						-
Date received and Date of initial service (Condition of receipt - new, used, etc.)	--		4.5.4		7.1	-
Current location				.63c		-
Details of maintenance						-
Date of last calibration (reports)						-
Maximum calibration interval						-
Label or tag with calibration date and due date						-
Calibration		5.3.3	4.9.2		9.0/9.6/	8/9++
Before use and to established program	7.1			.63	8.3	8.3
Traceable to National Standards, international standards or correlation inter-lab studies	7.2	--	--	.105		8.3.5
Reference standards with single use	7.3					8.3.6
Standards traceable to national or international standards	7.4			--/.107	9.6.3	8.3.6
Checks between calibration (where revealment)	7.5					8.4
Reference materials traceable to reference standards	7.6					8.3.7
Characterization of materials	--	--	--	--/.135		8.3.7

Comparisons of Standards
Page 4

Subject	25	45001	092	FDA/EPA#	2	MOE
Test Methods and Procedures:		5.4.1	4.8.1	.31e/.63b	6.3.4/	8.0/9.0++
Adequate documented instructions	8.1			.81	6.3.5/	8.1.1
Specifications available to staff	8.2				6.3.6	8.1.1
Non-standard methods fully documented	8.3					11.1
Checks of manual calculation and data transfer	8.4					
Control of electronic data processing	8.5					11.2.5
Subcontracted work	--	5.4.7	4.5.2		9.5/13.0	-
Environment		5.3.2	--	.41	11.0	5.0++
Controlled to not effect the test, staff, etc.	9.1			.43		5.1.2
Controlled access by outside persons	9.2					-
Good housekeeping	9.3					5.2
Handling of items to be tested.		5.4.5	4.4	.47	8.1/8.2	6.0++
System to identify samples/test items	10.1		4.8.1 4.7			6.4
Bonded storage, if necessary	10.2					6.3.1
Protection from damage	10.3					-
Receipt, retention and disposal procedures	10.4			.95		6.3.2
Records		5.4.4	4.9.4	.33f	6.3.7/8.4	11++
Suitable for repetition of test-	11.1		&	.51		11.1
original observations,			4.15	.130		-
calculations, calibration and final report				.195		11.2
Meet local law requirements	11.2					-
Secure and confidential	11.3					11.2.3
Comparisons of Standards	--	--	--	.190		-

Subject	25	45001	Q92	FDA/EPA#	2	MOE
Test Reports		5.4.3	4.1.4	.35b7/		10++
Accurate, clear and unambiguous on presentation of results and revealment information	12.1			.33a8/ .185		x
Include the following:	12.2					
Name and address of laboratory						x
Unique identification (serial No.) on all pages						x
Name and address of client						x
Description and identity of test item						x
Date of receipt of item and testing						x
Test results relate to item tested						x
Identification of method, procedure, specification, etc.						x
Description of sampling procedure						
Deviations, etc. from method						
Disclosure of non-standard methods used.						
Support data - charts, etc.						
Statement of measurement uncertainty						
Signature and title of technically responsible individual and date of issue of report			4.9.3			x
Statement about reproduction without approval						
No advice or recommendations						
Explanation of extrapolation from sample to lot						10.5(1)
Format of report controlled and standardized	12.3					10++
Control of correction (supplement to report)	12.4	--	4.12			11.2.4
Data Validation	--	--	--	--	10	10.3/10.4
Client cooperation	--	6.1	--	.10	1.3	3.3
Accreditation Body cooperation	--	6.2	--	.15##		3.6.1
Other lab cooperation (round robin)	--	6.3	--			-
Accreditation responsibilities	--	7.	--	.17###		-
Purchasing control	--	--	4.5	--		-
Incoming acceptance materials	--	--	4.5.5		7.0	-
Client supplied product	--	--	4.6			-
Special processes	--	--	4.8.2			-
Receiving inspection	--	--	4.9.1			6++
Test status	--	--	4.11	.35b1		-
Internal audits	--	--	4.16	--	18	3.6
SPC	--	--	4.18	--	15	-
Reagent control	--	--	--	.83		-
Economics	--	--	--	--	1.4/19	-

FDA/EPA do not accredit. It addresses audit, response, etc. .200 also addressed this subject related to legal actions.

Effects of non-compliance

(1) Provision of Data Interpretation

CODE OF PRACTICE/ DOCUMENT(S) ISE

- Definition
- Application
- Content
- Sources

STANDARDS OF INTEREST TO LABORATORIES

*	ISO Guide 25	General Requirements for the Technical Competence of Testing Laboratories
	ISO Guide 38	General Requirements for the Acceptance of Testing Laboratories
*	MOE	Code of Practice for Environmental Labaoratories
*	CSAZ201-M1990	Qualification Code for Ontario Laboratories analysing Industrial Waste
	EN 45001	General Criteria for the Operation of Testing Laboratories
*	ANSI/ASQC Q2	Quality Management & Quality Systems Elements for Laboratories - Guidelines

Under Construction:

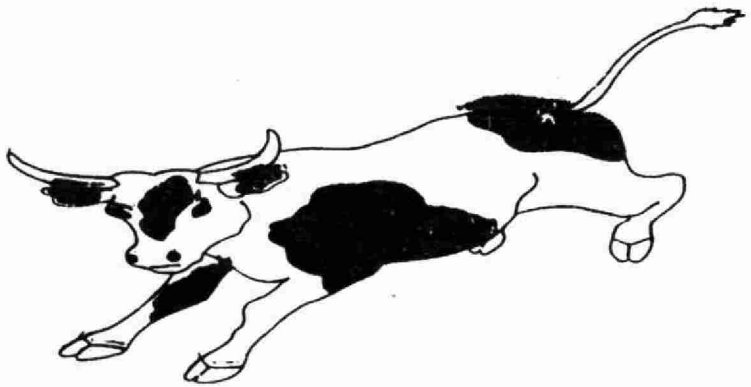
MOE/CAEAL	Practical Guide for Laboratory Analysis of Environmental Samples
EPA	(DRAFT) Good Automated Laboratory Practices Recommendations for Ensuring Data Integrity In Automated Laboratory Operations with Implementation Guidance

Mailing List

Documents	Contact(s)
CSA ISO	Canadian Standards Association Consolidated Mailing List 178 Rexdale Boulevard Rexdale, Ontario Ph (416) 747-4368 or Quality Management Institute (A Division of CSA) Mississauga Executive Centre Suite 1420 4 Robert Speck Pkwy Mississauga, Ontario L4Z 1S1 Ph (416) 272-3920 FAX (416) 272-3942 (Note: CSA and QMI are Canadian Distributors for ISO)
MOE	Ministry of Environment Laboratory Services Branch Quality Management Office P.O. Box 213 Rexdale, Ontario M9W 5L1 Ph (416) 235-5758 FAX (416) 235-6110
USEPA	United States Environmental Protection Agency Office of Information Management Research Triangle Park North Carolina 27111
ANSI/ASQC	American Society for Quality Control Customer Service P.O. Box 3066 Milwaukee, Wisconsin 53201 Ph (800-248-1946 TOLL FREE)

WHAT WAS *NIMMP?*

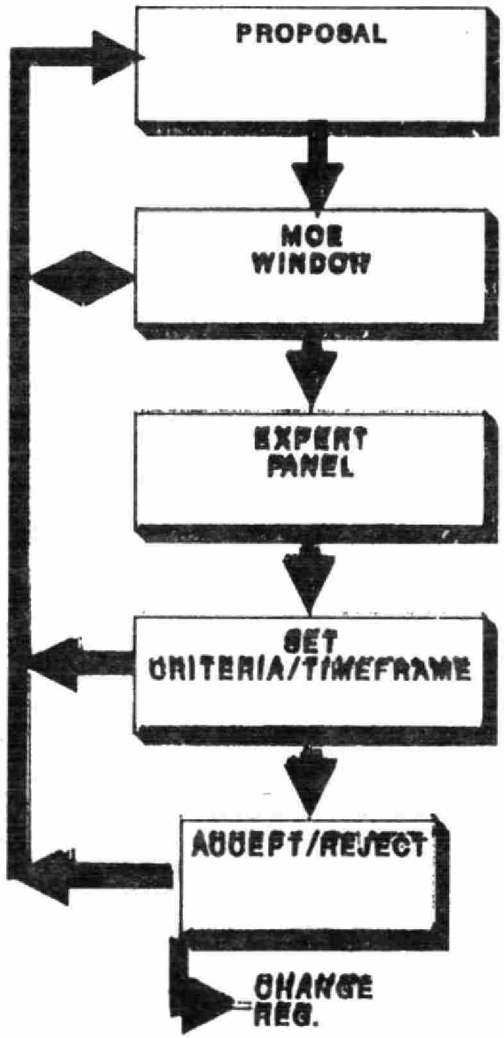
N NEW
I INSTRUMENTAL
M MEASUREMENT
M METHOD
P PRINCIPLES



PRINCIPLES & PROCEDURES
"HOW", NOT WHAT OR WHY!

- NIMMP MAY EXPAND TO ENTIRE MISA REGS
- BUT ONLY TO IMPROVE THE "HOW"
- WHAT AND WHY ARE ADDRESSED BY REGULATION-SETTING OFFICE
- PRINCIPLES = A FEW KEY WORDS
- PROCEDURES = DETAILED SPECIFICATIONS

WHAT WILL HAPPEN?



PROPOSAL

MUST SHOW IDEA:

- "WORKS" BEYOND A REASONABLE DOUBT
- MEETS THE MDL (ANALYTICAL)
- BENEFITS USERS OR GOVT.

MOE WINDOW

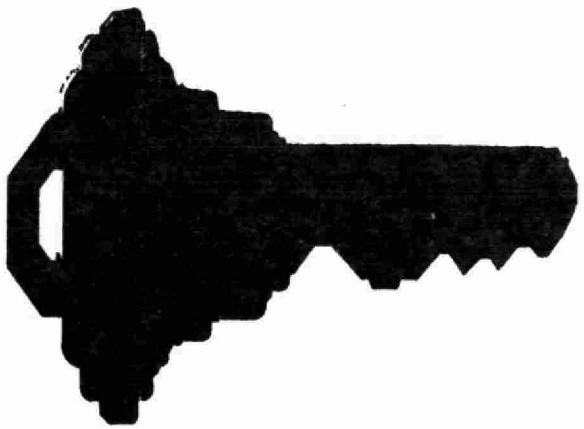
- ONE CONTACT TEL. NO. & ADDRESS
- WINDOW SETS UP EXPERT PANEL
- KNOWS STATUS OF PROPOSALS
- CLOSES FILES WHEN PROPOSAL IS ACCEPTED OR REJECTED
- PASSES APPROVED PROPOSAL TO REGULATION TEAM

EXPERT PANEL

- EXPERT(S) WILL REVIEW PROPOSAL
- "PANEL" WILL SET CRITERIA & TIMEFRAME
- "PANEL" WILL WORK WITH PROPOSER
- ACCEPT/REJECT PROPOSAL WITH REASONS
- REPORT BACK TO WINDOW AND PROPOSER

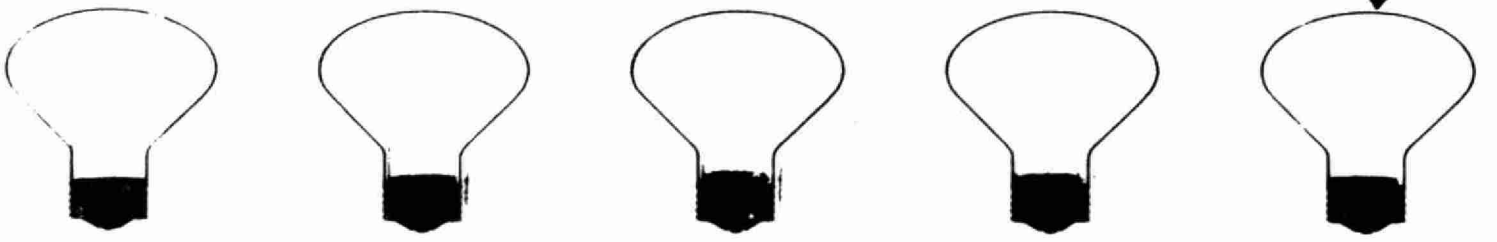
CHANGE REGULATION(S)

- PRINCIPLES AND PROCEDURES
- EXISTING REGULATIONS -SLOW
- NEW REGULATIONS -WHEN THEY COME
- MOE STRIVES TO RETAIN FLEXIBILITY



CHANGES TO NIMMP
(UNDER DISCUSSION!)

- EXPANDING AND GENERALIZING ROLE ✓
- ONE WINDOW -GUARANTEED RESPONSE ✓
- EXPERT PANEL (GUIDANCE DOCUMENT) ✓
- DEVELOP CRITERIA AND TIMETABLE WITH PROPOSER ✓
- SIMPLIFIED EVALUATION PROCESS ✓



ELECTRONIC COMMUNICATION

What's New At The MOE Lab? ...

ELECTRONIC COMMUNICATION

Will affect how we work by ...

- Allowing new types of task structures.
- Fostering new types of reporting relationships.
- Promoting a more democratic forum where ideas can be exchanged spontaneously and casually.

ELECTRONIC COMMUNICATION

An Electronic Bulletin Board Could Be Used For....

- Messages
- Exchanging Files
- Peer Review of Papers, Methods, Presentations
- Asking "Does Anybody Know...? questions.
- Bulletins Announcing Upcoming Events.

ELECTRONIC COMMUNICATION

What is Required? ...

- Computer; any computer with a communication port.

- Modem; any modem. HAYES or HAYES-compatible are easiest to work with:
  2400 baud is maximum transmission rate at this time.

- Software; Any software that will handle the modem. (e.g. PC-DIAL, PC-TOOLS, TELIX, WINDOWS ETC.)

ELECTRONIC COMMUNICATION

MOE Quality Management BBS...

- The MOE QM BBS is currently being run in the QM Office.

- It was developed to:
  - allow users to obtain MOE analytical methods
  - act as an "information exchange center" for Lab, MISA and QM issues between MOE and clients

- Will reduce paper usage, postage costs, time and money

ELECTRONIC COMMUNICATION

How Do We Access The BBS? ...

- Dial (416) 235-6136 from your computer to log on the MOE QM BBS.

- Follow the menu to download MOE analytical methods, upload information, read the latest LSB/MISA bulletins, or leave a message/comment.

- The System Operators are:
  -Cammy Lynn Raposo (Voice: (416) 235-6005)
  -David Boomer (Voice: (416) 235-5858
  Spec. BBS: (416) 235-6119)

ONTARIO MINISTRY OF THE ENVIRONMENT
ITC SECTION

Automation Of Both;

Quality Control
And
Quality Assurance

TOPICS & GOALS

- Topics:
  - History
  - Concepts Of QA And QC
  - Describe a tool for the automation of quality control.
  - Describe a tool for the automation of quality assurance.

- Goals:
  - Provide insight w.r.t. how we got here.
  - Highlight some important implications associated with how we define QA and QC.
  - Show how PC's, and PC networks can be used to automate both QC and QA procedures.

?? WHY AUTOMATE ??

WHY AUTOMATE ?

- QUALITY CONTROL
  - ▸ Reduce Personal Error
  - ▸ Ensure That All Procedures Occur As Specified
  - ▸ High Level Of Customization Associated With METHOD Not Technician
  - ▸ Higher Productivity

- QUALITY ASSURANCE
  - ▸ Integrate Complex Functions
  - ▸ Standardized Procedures For Data Review & Validation
  - ▸ Summary Reports From RDBMS

----------->>> QUALITY IS FREE!!!

Brief History:

- ICP X-Ray Automated Techniques; Generated large sets of data requiring computation and processing.
- 6502 Boards, CBM/PET TRS-80 etc
- Productivity Up >300%...
- Problem/Challenge!!

  Every Time a change in DCI or QC protocol occurred we were faced with the 'Opportunity To Grow' that was associated with reprogramming ALL of the different systems!!

SOLUTION ? !

A NETWORK Of Computers!!!

- A Single Program For DCI !!!

... Also provided services like electronic mail,
Wordprocessing, Spreadsheet, DataBase,
Presentation Graphics, Etc

NETWORK:

QA/QC Implications

- All Data Through One Wire
- All Data Stored In One DataBase
- ... Obvious QA/QC Possibilities!!

KEY CONCEPT:

QA is Separate From QC !!!

- QC occurs in 'Real Time' and is directed at reducing the effect of Determinate Error to levels where the system appears to be under the influence of random variation; ie QC procedures should be linked to known sources of determinate error.

- QA occurs 'after the fact' and is directed at providing documentation that attests to the fact that QC procedures were well designed and were carried out in an appropriate manner.

QA is Separate From QC !!!

- Implications w.r.t. ;
  - Division Of Labour
  - Assignment Of Responsibility
  - Assignment Of Authority
  - Allocation Of Resources
  - Conceptualization Of System

QC-EXPERT

LQAS

QA & QC AUTOMATION

- QC: { Many Custom Systems; One For Each Analytical System }
  - ► linked to sources of determinate error
  - ► specific to METHOD
  - ► real-time on real systems
    - ✓ Therefore, not easily supported on LIMS
  - ► "QC-EXPERT" is one example of an automated QC system for spectroscopic instruments.

- QA: { One Central System On PC-LIMS }
  - ► links and summarizes all QC results
  - ► acts after-the-fact
  - ► does not have to be customized, as with QC
  - ► LQAS is an example of an automated QA system

QC-EXPERT

- Developed in the 70's for use on ICP/OES systems using PET computers.
- Extended during early 80's to ICP/MS using PC's, dBase, and LOTUS.
- Extended further during 80's to include QA database on PC network.
- 'Collaboration' with Perkin–Elmer and BMB to become a commercial product.

QC-EXPERT

- Using a sophisticated autosampler, QC–Expert...

√ Controls the instrument & autosampler.
√ If data is out of spec, QC–Expert carries out predetermined procedures to restore quality.
√ Creates text and graphic reports summarizing quality control results.
√ Summary reports are sent to the LQAS system.

QC-EXPERT

Limits Can Be Associated With...

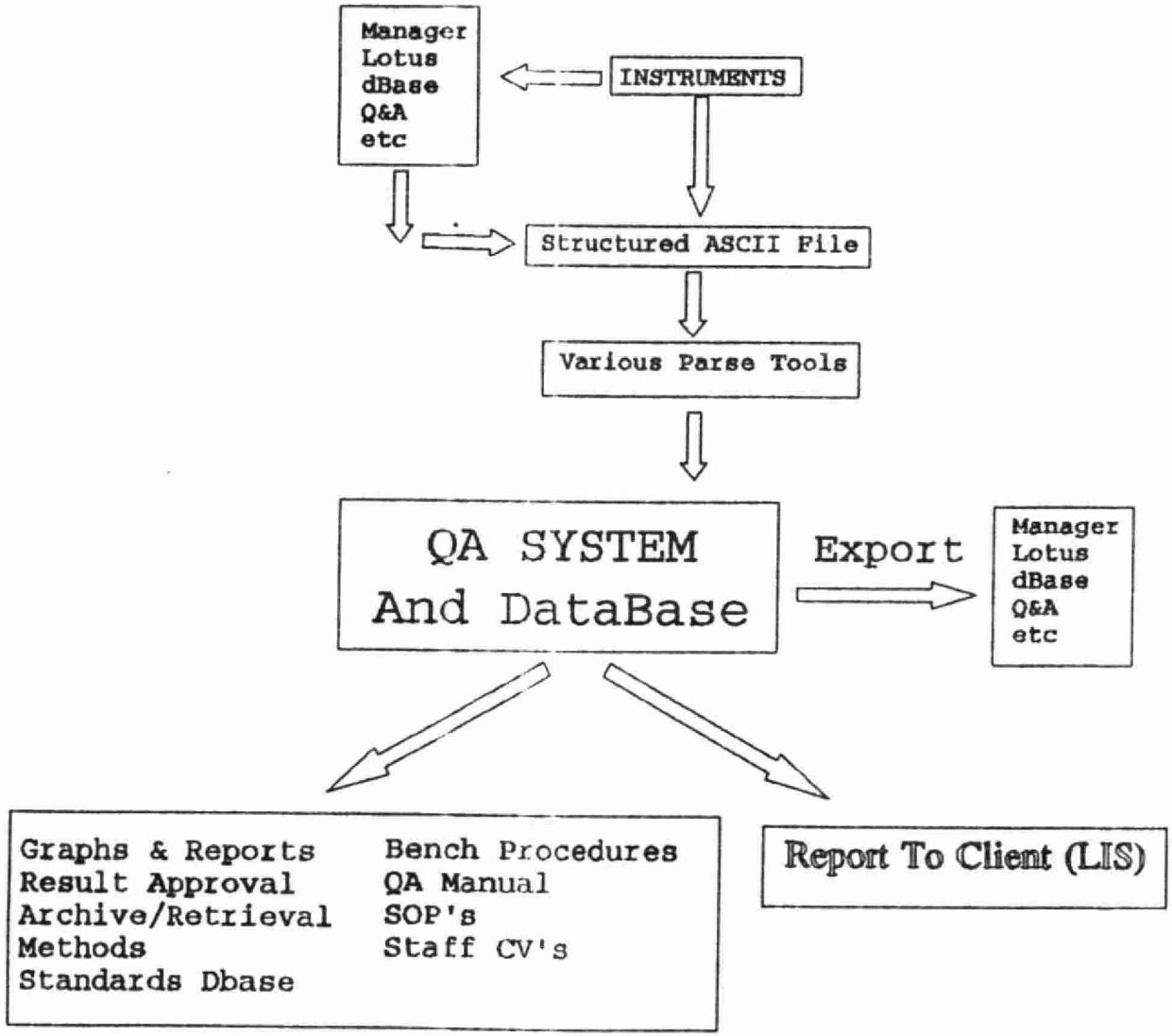
- **Standardization Coefficients**
- **Correlation Coefficients**
- **QC Standards And Blanks**
- **Internal Standards**
- **Duplicates**
- **Spikes**
- **Dilutions**
- **Samples**

QC-EXPERT

Actions May Be...

- **Print Message And Stop**
- **Print Message And Continue**
- **Restandardize And Rerun Or Continue**
- **Restandardize And Rerun**
- **Wash For Specified Time And Rerun Or Continue**
- **Rerun Current Sample/Standard**
- **Continue From Specified Position**
- **User--Defined Program**

PC BASED LAB QA SYSTEM: LQAS



LQAS

- **Functions**
  - √ Data Capture (From QC–Expert)
  - √ Results Approval
  - √ Reports And Graphs
  - √ View And Update; Methods, Bench Procedures, QA Manual, SOP's, CV's, Standards Databases, Archive And Retrieval.

- **Will run on LIMS (DOS) or on stand–alone computer.**

DATA CAPTURE

RESULTS INSPECTION AND APPROVAL

RESULTS APPROVAL

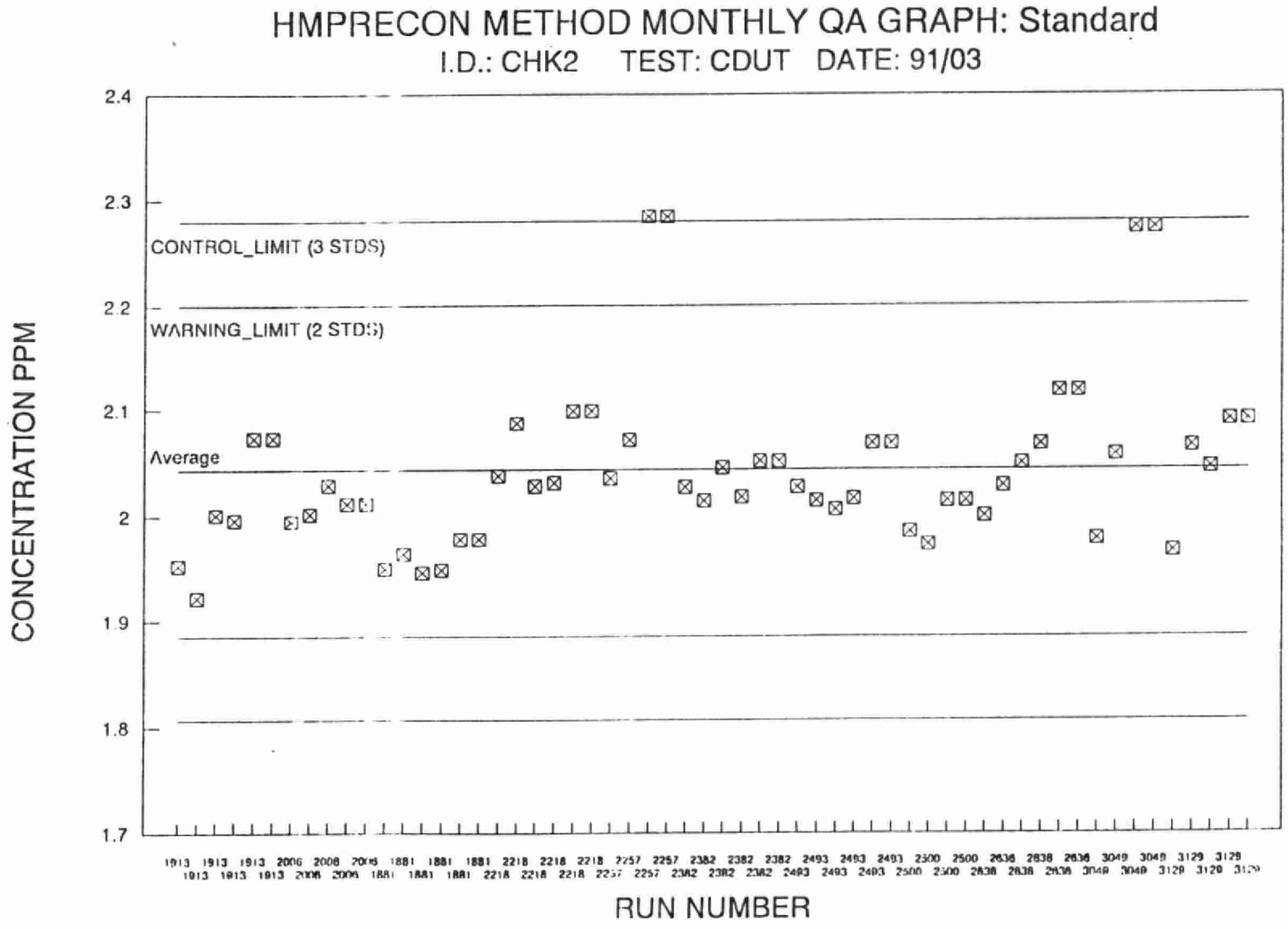
- Automated QA Inspection And Approval... Report via E-Mail

- Manual QA Inspection And Approval

  Both Procedures Are Supported By The LQAS "Tracking System"

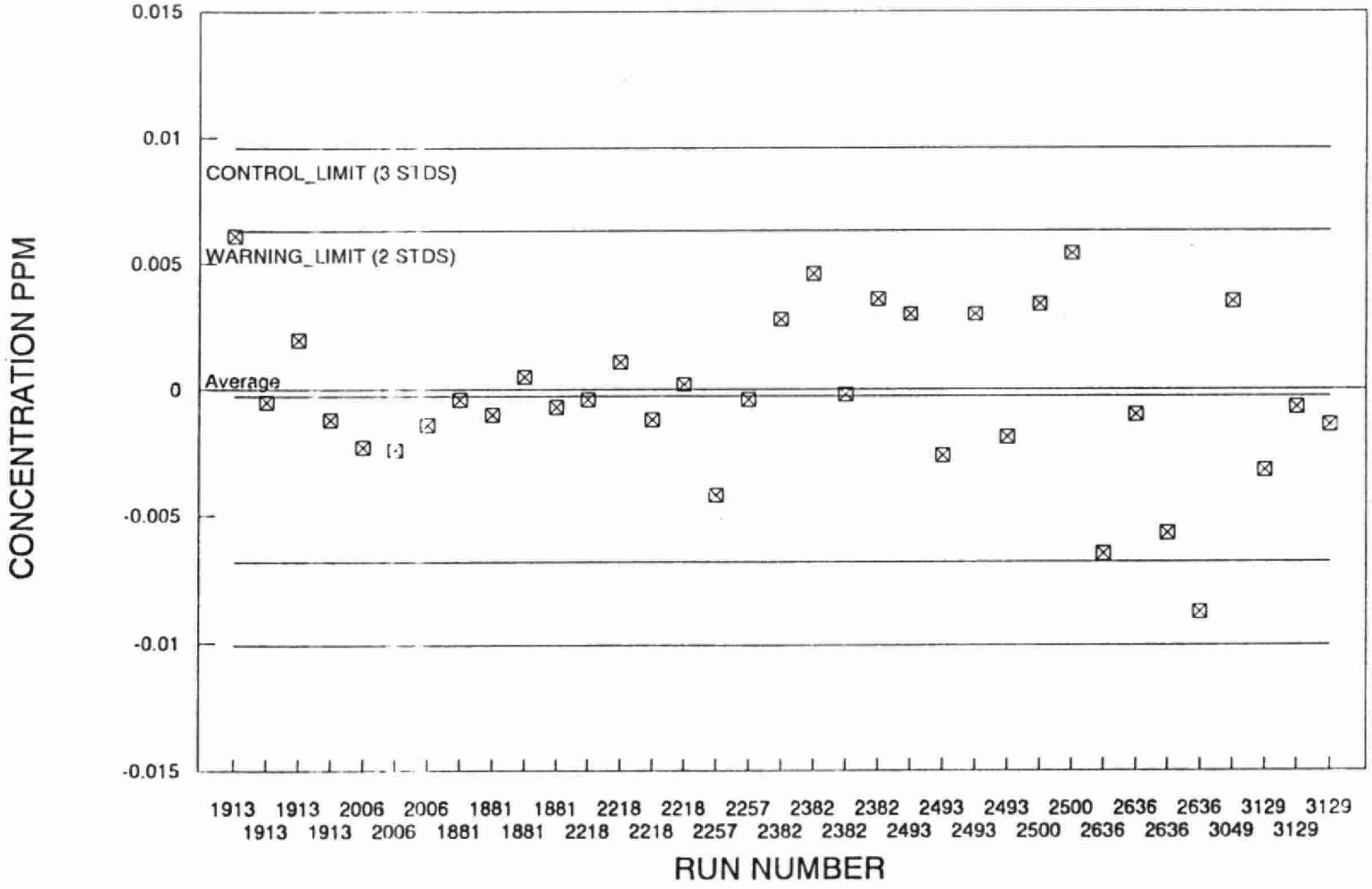
REPORTS AND GRAPHS

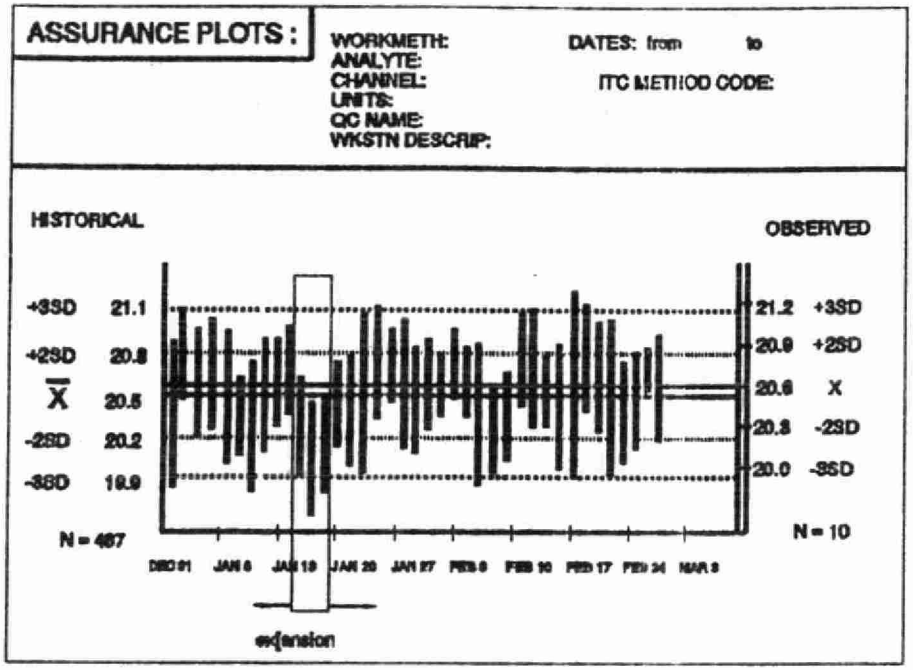
25


HMPRECON METHOD MONTHLY QA GRAPH: Standard
I.D.: CHK2 TEST: CDUT DATE: 91/03
CONCENTRATION PPM
2.4
2.3
2.2
2.1
2
1.9
1.8
1.7
CONTROL_LIMIT (3 STDS)
WARNING_LIMIT (2 STDS)
Average
1913 1913 1913 2006 2006 2006 1881 1881 1881 2218 2218 2218 2257 2257 2382 2382 2382 2493 2493 2493 2500 2500 2636 2636 2636 3049 3049 3129 3129
RUN NUMBER

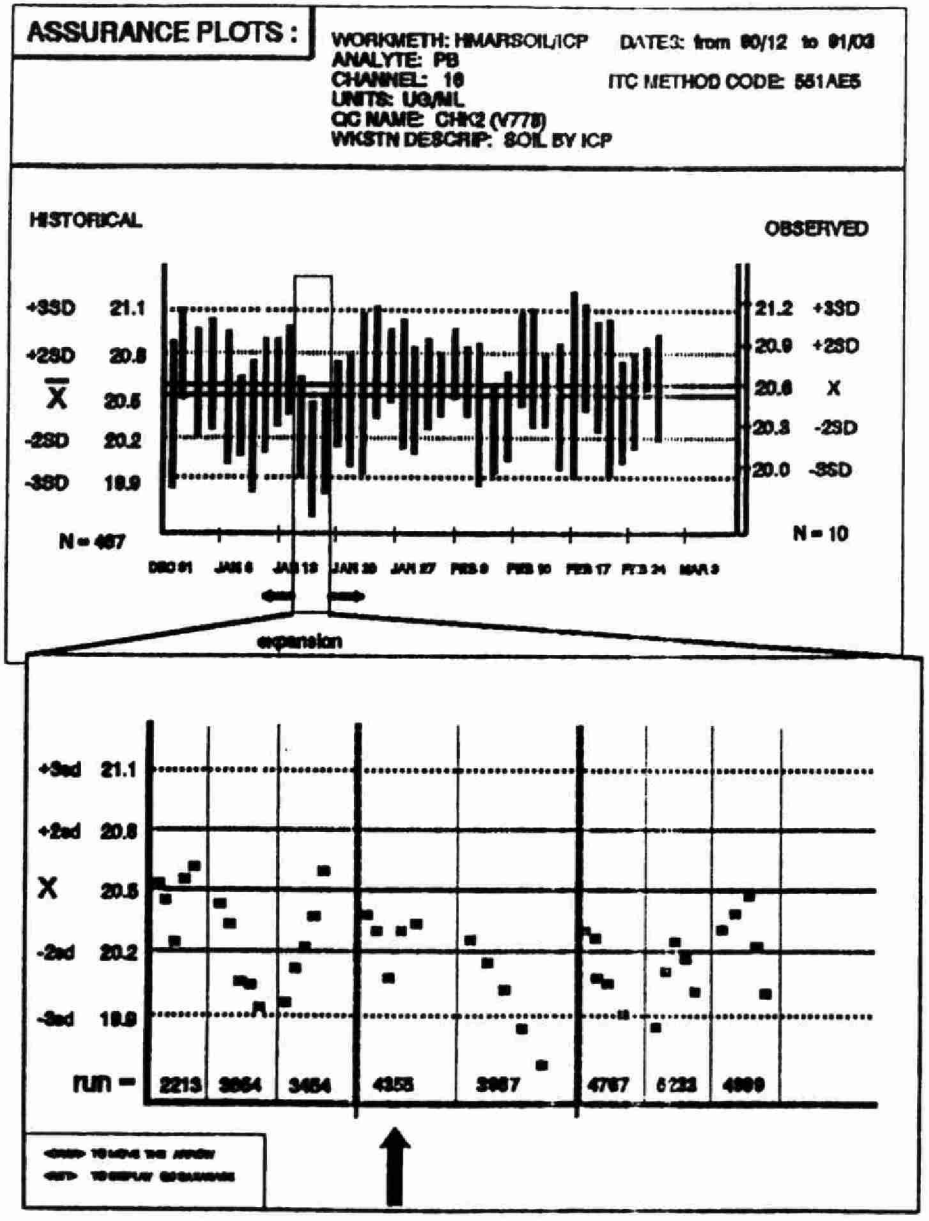
HMPRECON METHOD MONTHLY QA GRAPH: Blank

I.D.: BLK TEST: CDUT DATE: 91/03

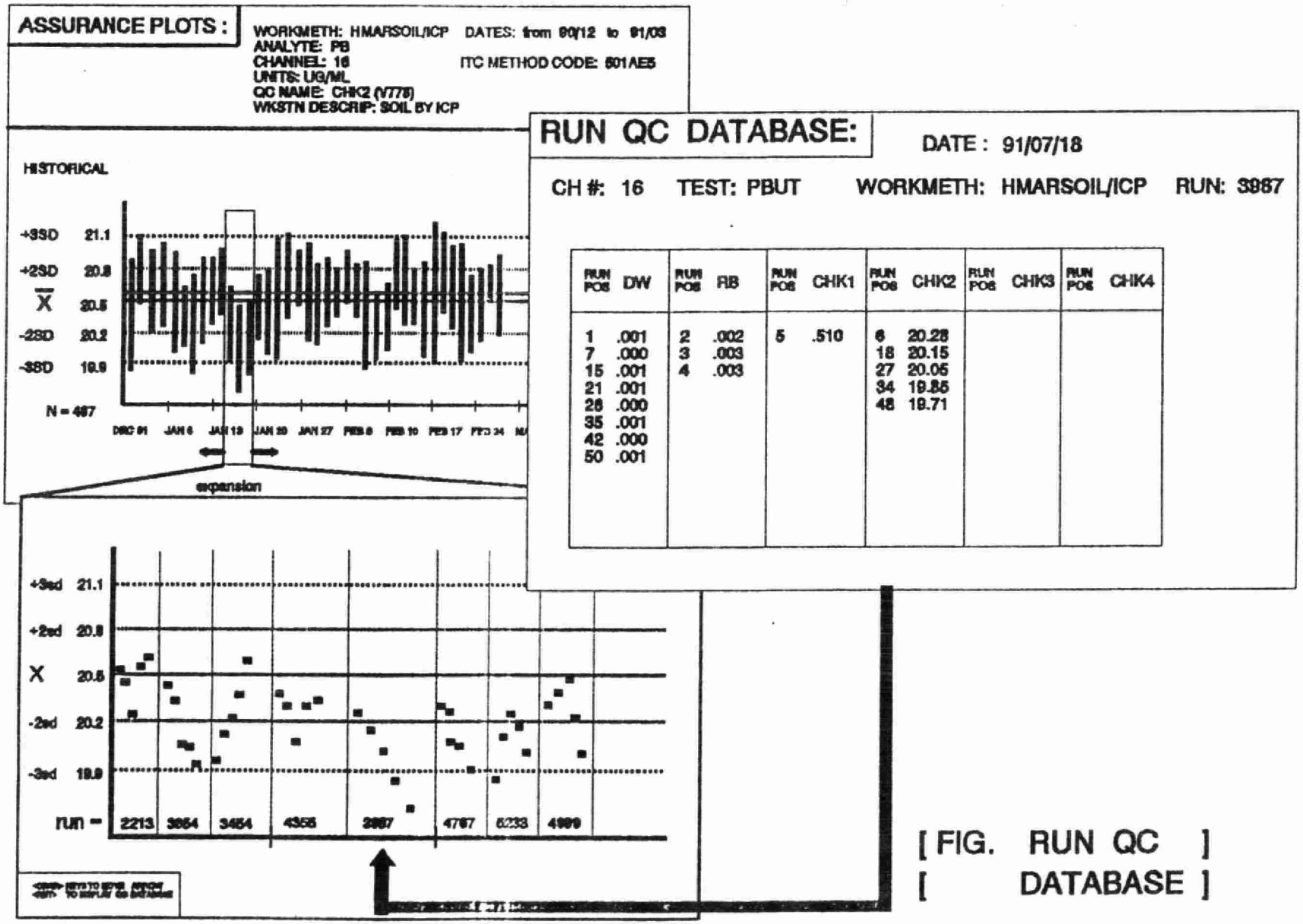




[FIG. ASSURANCE PLOTS]



[FIG. ASSURANCE PLOT]
[EXPANSION]



[FIG. RUN QC]
[DATABASE]

"Quality is never an accident. It is always the result of high intention, sincere effort, intelligent direction, and skillful execution.

It is the wise choice of many alternatives, requiring the combined knowledge of many masters of craftsmanship.

Quality is not an attribute; it is a process, an ideal that can only be realized after the demands of necessity have been met and afte the attainment of mere usefulness"

Willa Foster

JB

SECTION THREE - Day 1 - Session Two Presentations

SECTION THREE

DAY 1 - SESSION TWO PRESENTATIONS

CHAIRPERSONS -	J. LEARN, DOMTAR C. RAPOSO, MOE
ROUND ROBINS (MOE) -	S. CUSSION, MOE
ROUND ROBINS (COMMERCIAL LAB) -	J. SZEKELY, HAMILTON REG. B. FOWLER, AXYS LAB
ROUND ROBINS (WTC) -	M. FOROUTAN, WTC P. FOWLIE, WTC
REGS & JAWG -	DR. O. HERRMANN, HYDRO

INTERLABORATORY STUDIES --

THE MOE LABORATORY PERSPECTIVE

SYLVIA CUSSION
QUALITY MANAGEMENT OFFICE, LSB

PURPOSE:

Performance evaluation technique

in support of Ministry Programs

PROGRAMS

- MISA
- Special (N-nitrosodimethylamine)
- Air Programs

MATRICES

- Ampouled Standards for Direct Instrumental Analysis
- Spiked Reagent Water
- Spiked Effluents
- *PUF* Cartridges
- HIVOL Filters

PARAMETERS

CONVENTIONALS/INORGANICS

- Metals
- TKN & Total Phosphorus
- Phenolics by 4AAP
- Oil & Grease
- Total Cyanide
- Mercury
- DOC & TOC

PARAMETERS

ORGANICS

- Volatiles
- Acids (Phenolics)
- Base/Neutrals
- Neutral Chlorinated
- BTX & Acrylonitrile
- N-nitrosodimethylamine (NDMA)
- Dioxin

EVALUATION TECHNIQUES

- Statistical Summaries
- Percent Recovery of Target
- Bar Graphs - Elution Order
- Youden Plots
- Automated Graphical Procedures for Rapid Evaluations of Interlaboratory Studies

STATISTICAL SUMMARIES

- Interlaboratory Mean
- Interlaboratory Median
- Range
- Standard Deviation

PERCENT RECOVERY

- Use Target/Design value

- May be affected by method of quantitation (eg. RFA Method)
  - Quantitate relative to one parameter (eg. DHA)

EXAMPLE OF RECOVERY CORRECTION - DHA

- RFA Method Quantitates all other parameters relative to Dehydroabietic Acid (DHA)
- Difference between laboratories attributable to difference in DHA
- Normalize parameters relative to DHA
- Re-evaluate performance of other parameters

FIGURE 11: INTERLABORATORY STUDY 88-2A

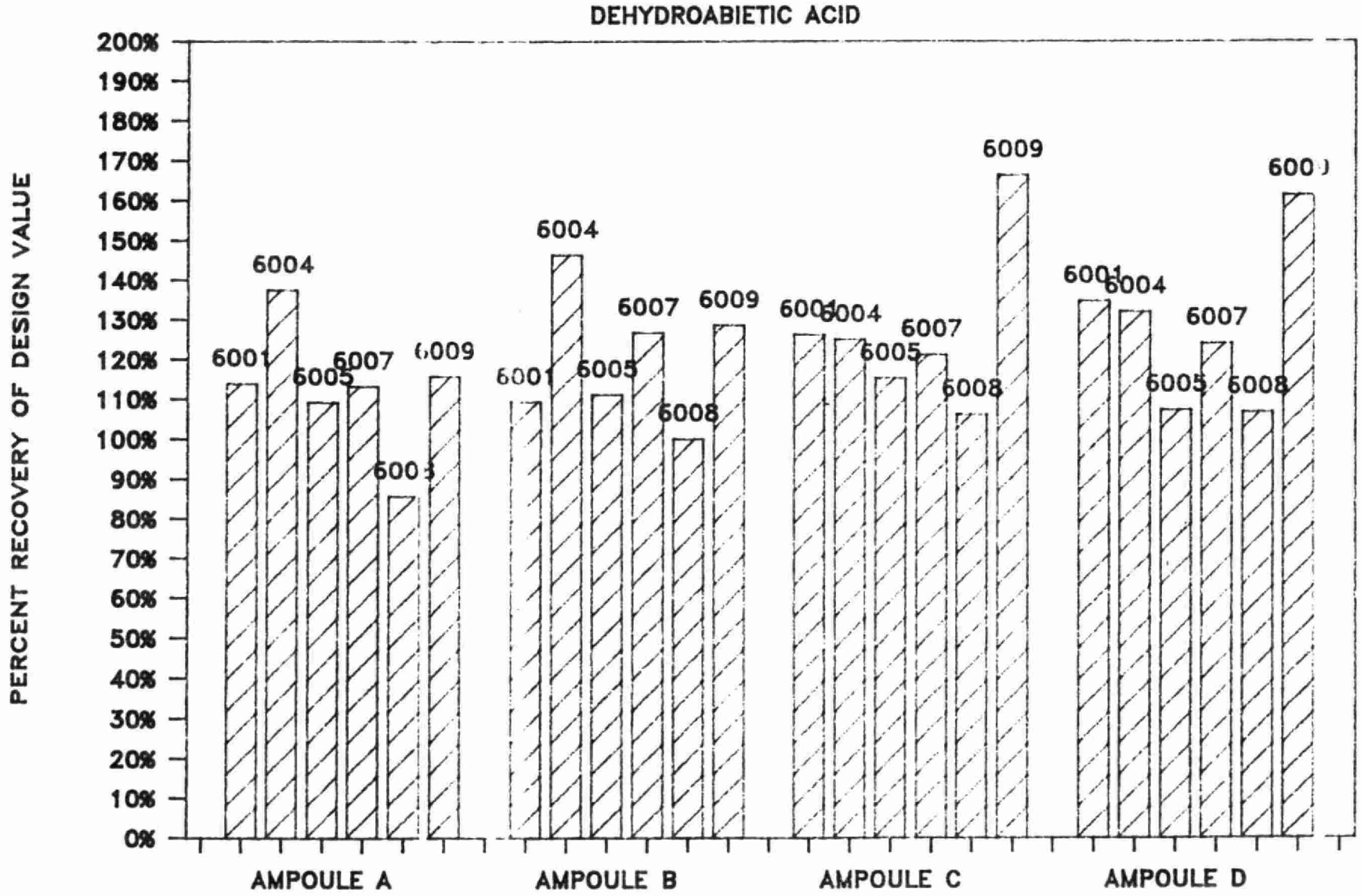


FIGURE 3A: INTERLABORATORY STUDY 88-2A

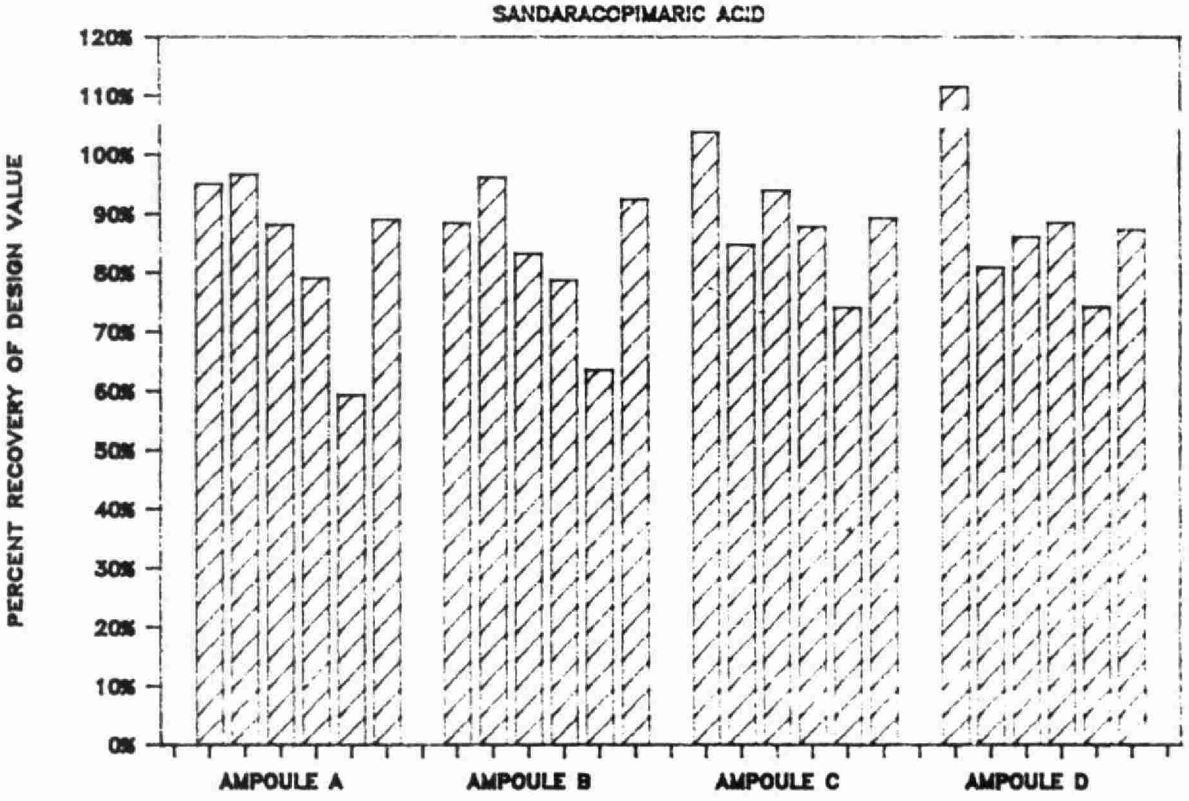
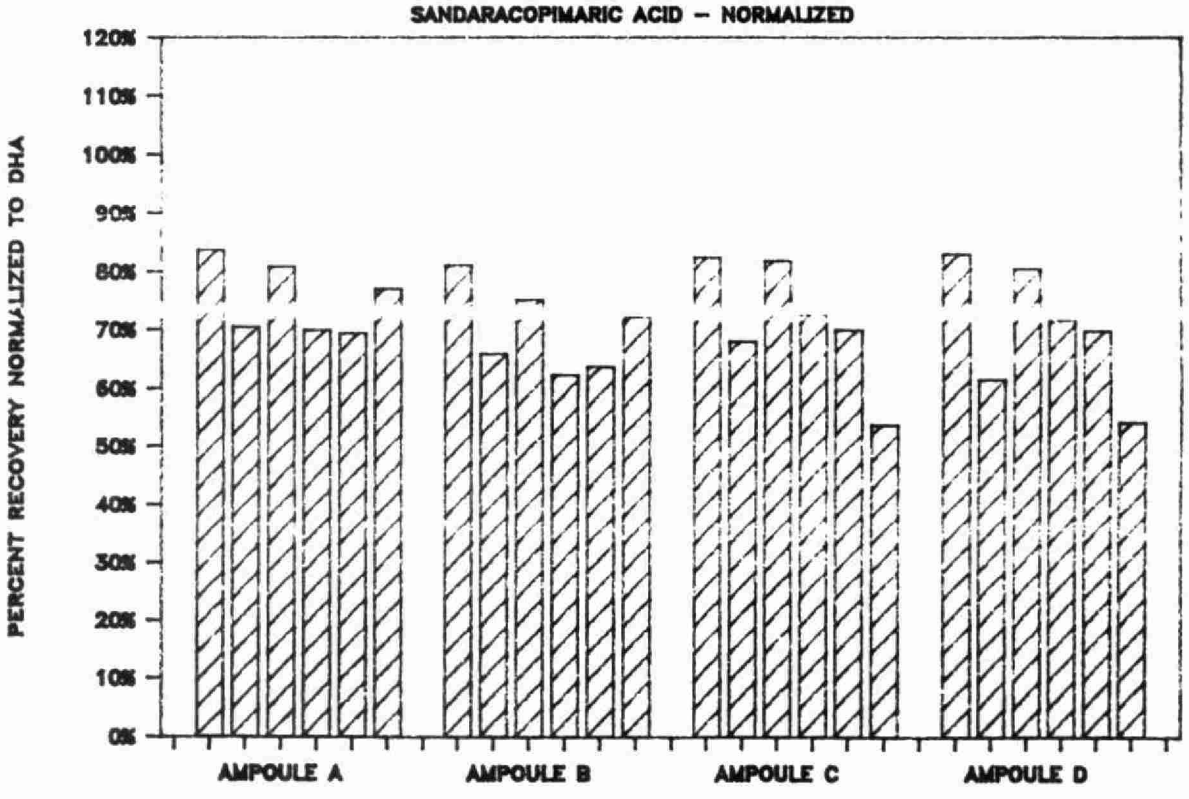


FIGURE 3: INTERLABORATORY STUDY 88-2A



BAR GRAPHS - ELUTION ORDER

- Organic Scans
- Base order on most commonly used Gas Chromatographic Capillary Column

FIG 1: ROUND ROBIN 88-1; VOLATILES

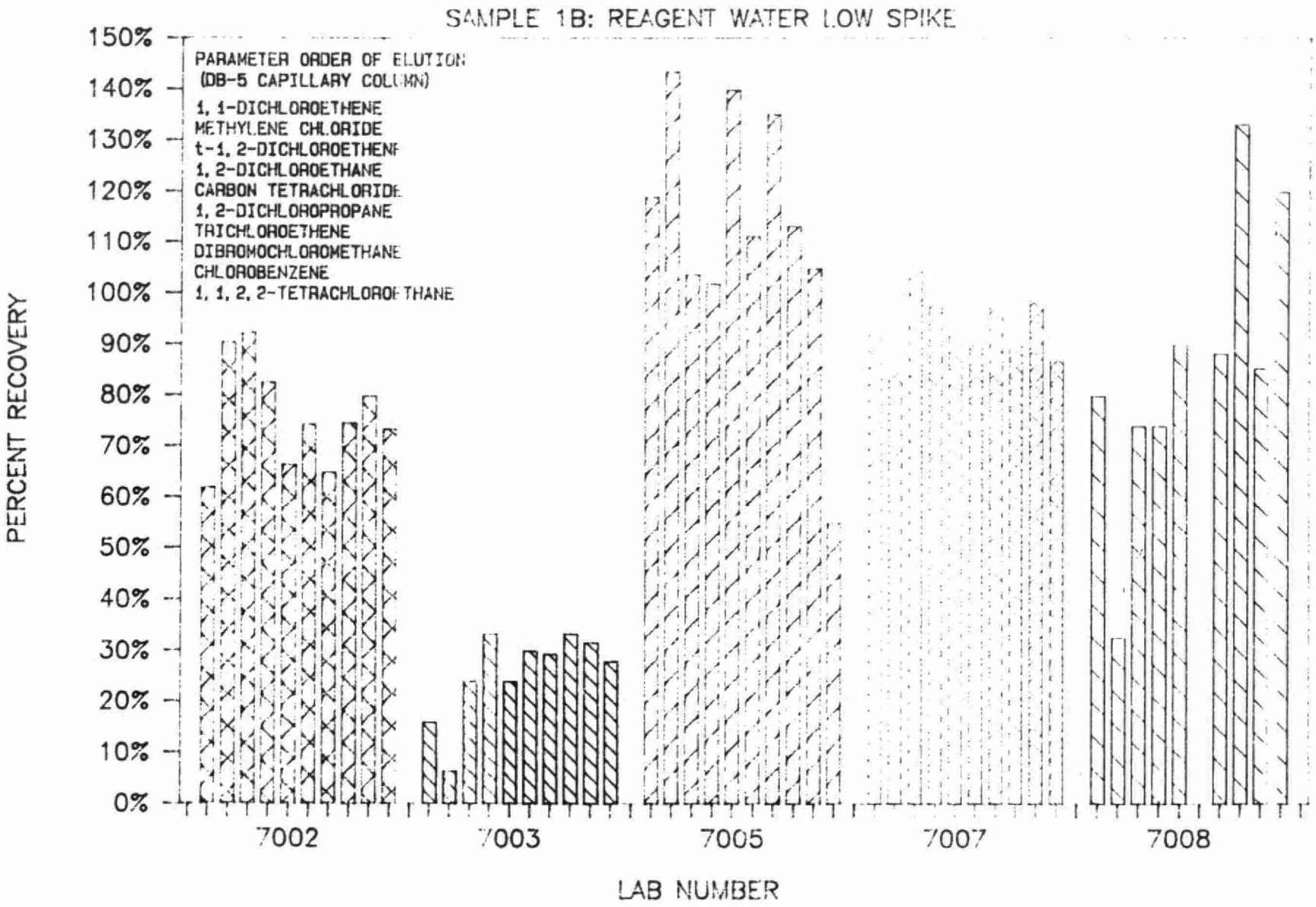
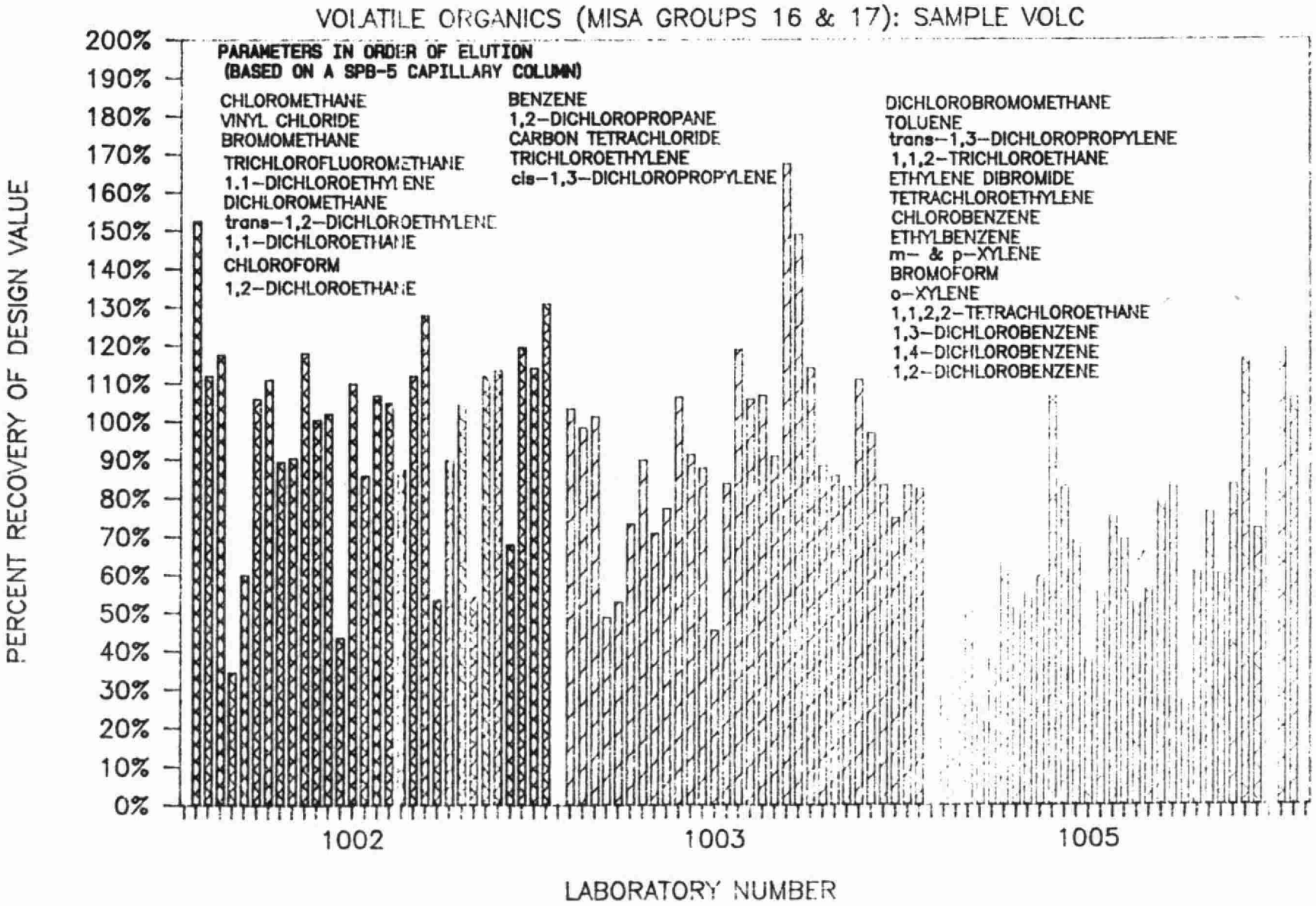


FIGURE 5 -- INTERLABORATORY STUDY 89-1



YOUDEN PLOTS

- Plot 1 sample on x-axis and 2nd sample on y-axis
- Expect results to cluster around target values
- Results along line passing through origin & target demonstrate bias
- Results in upper left & lower right quadrants are erratic - out of control

FIGURE 10 – INTERLABORATORY STUDY 89-1

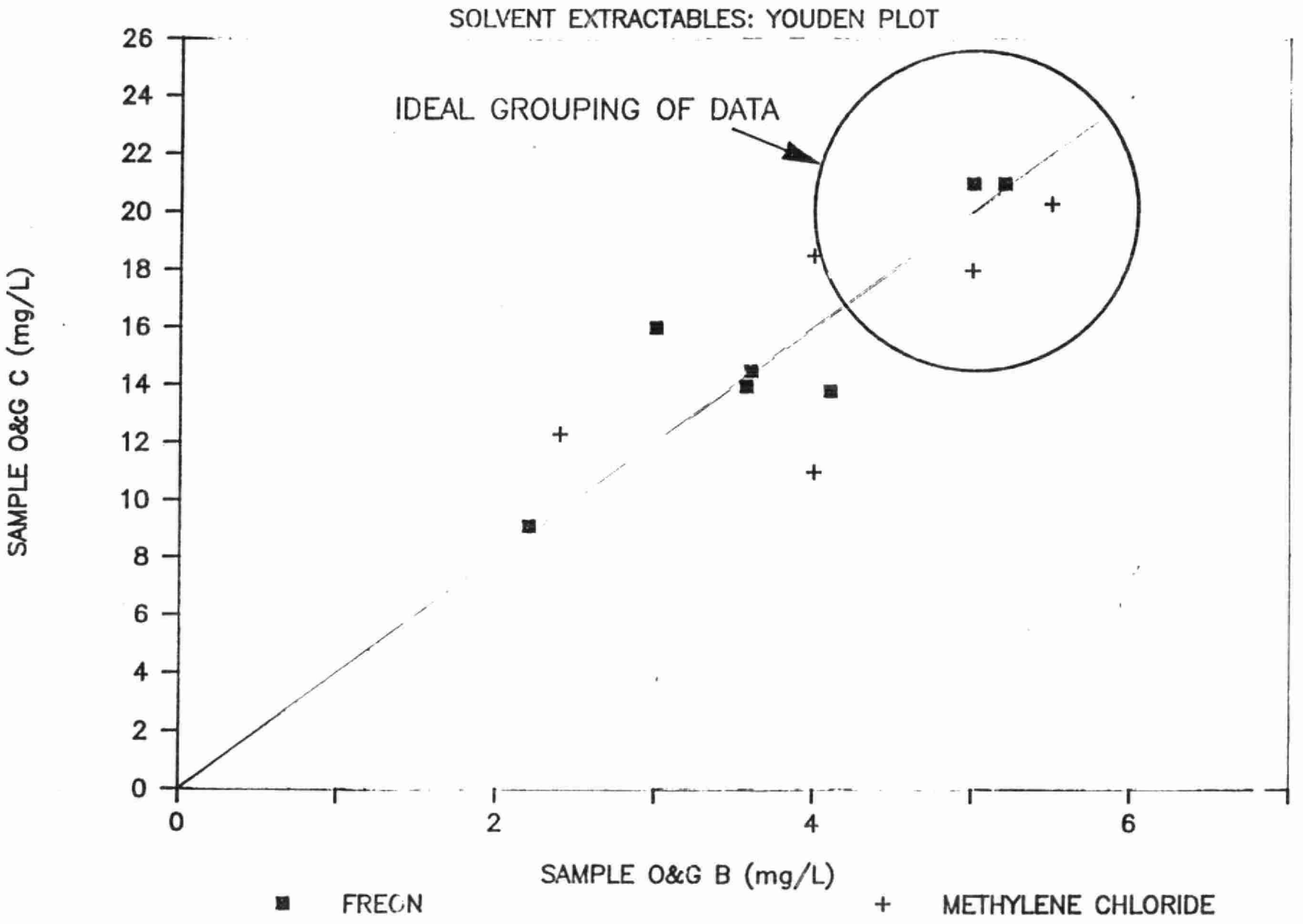
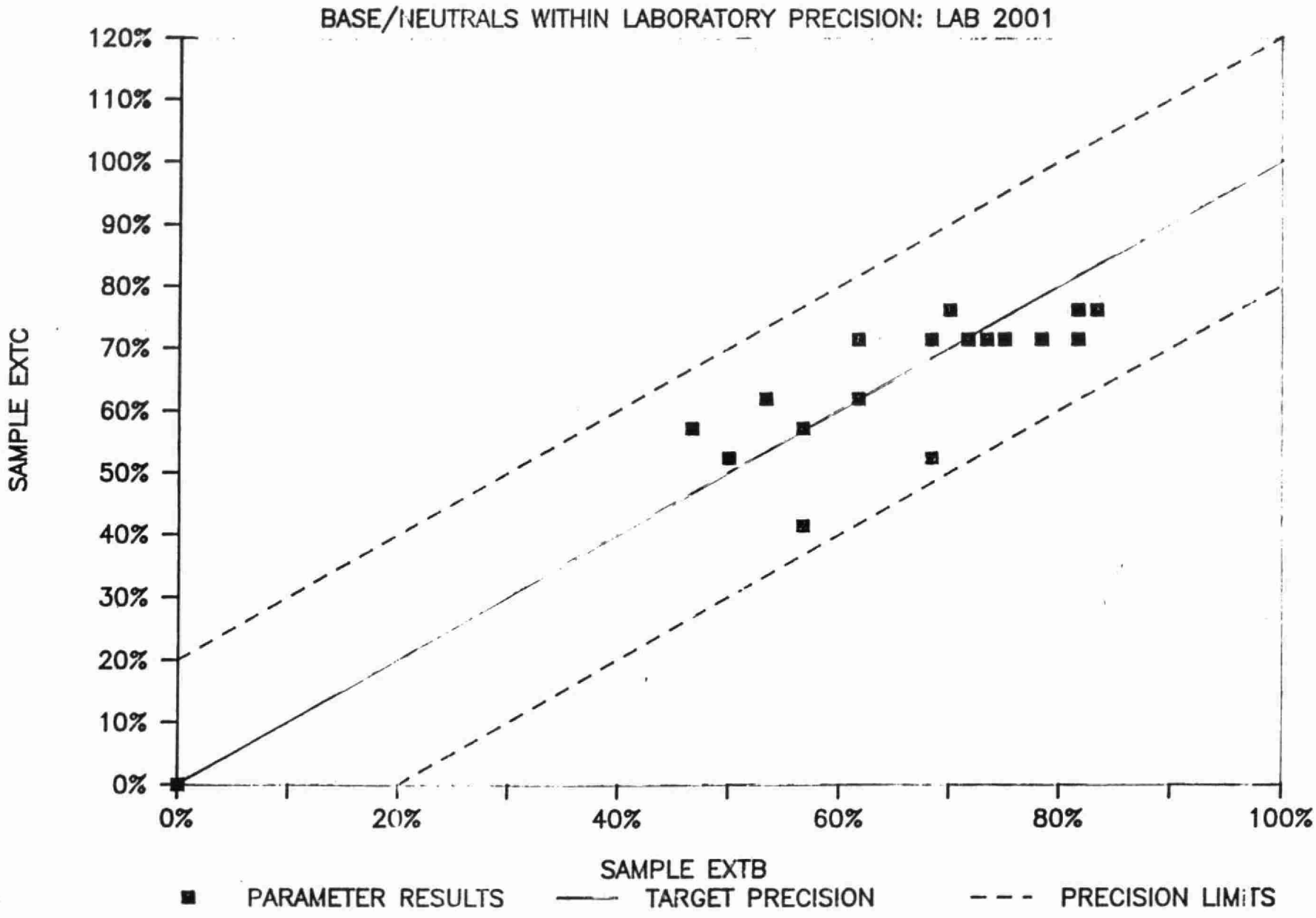


FIG. 9: INTERLABORATORY STUDY 89-5



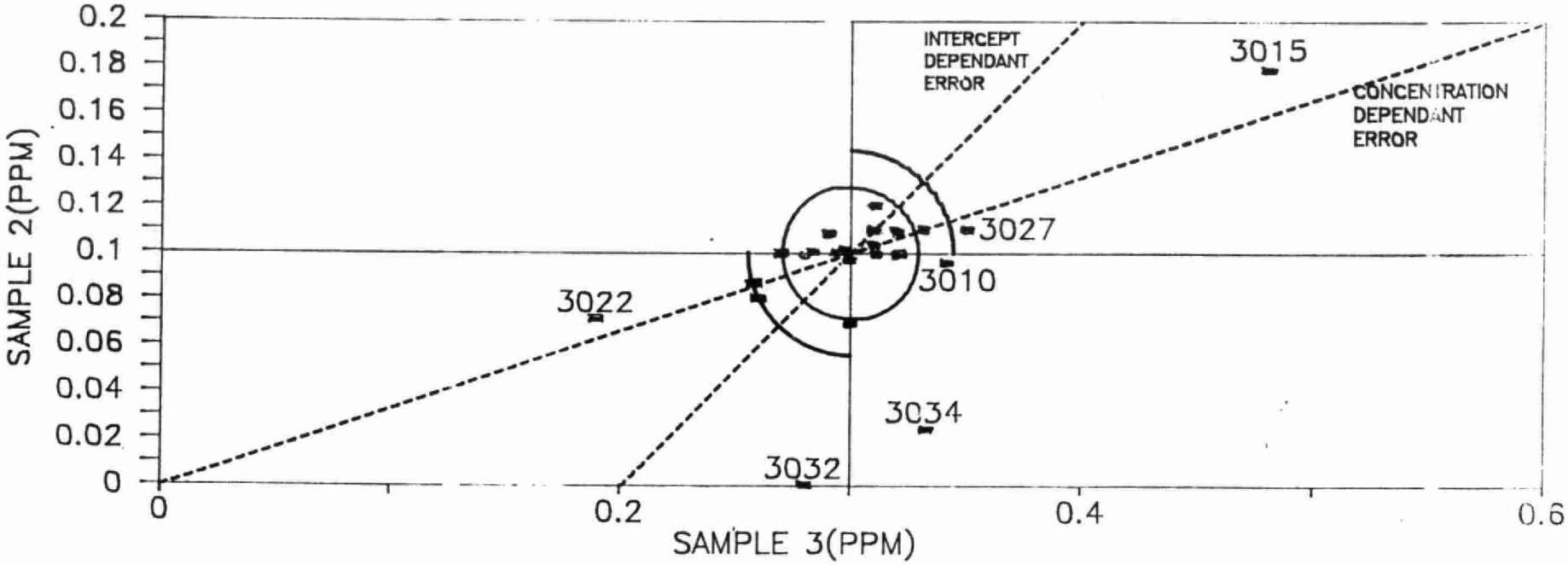
AUTOMATED GRAPHICAL PROCEDURES

- Rapid evaluation of interlaboratory studies
- Poster session in TRENT/*KAWARTHA* rooms (Sathi Selliah)
- Use to evaluate inorganics and organics

FIGURE 5.6

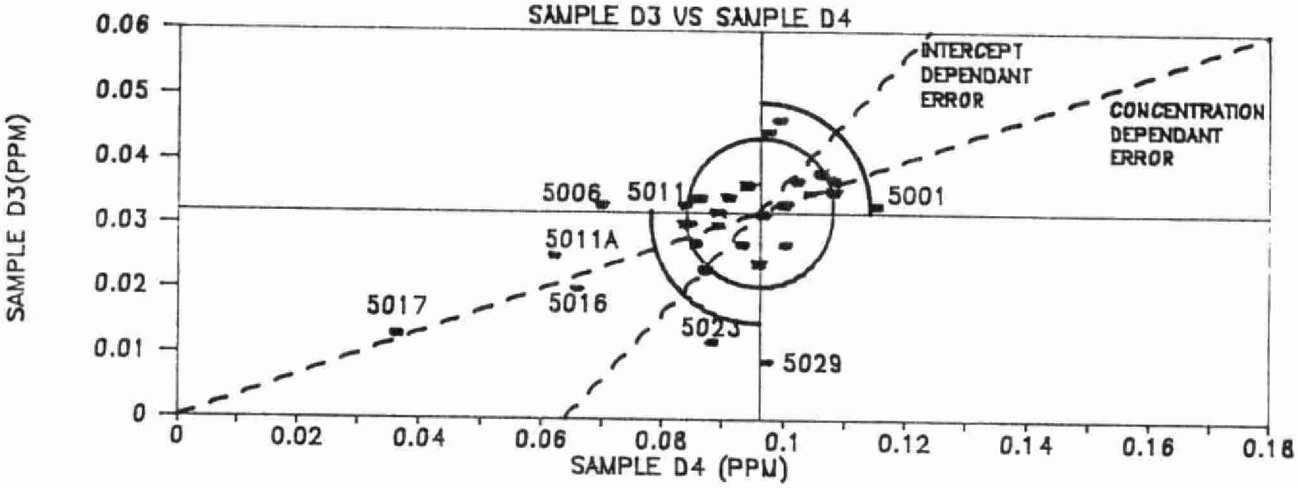
CHROMIUM – STUDY 89–6

INTERLABORATORY PRECISION EVALUATION



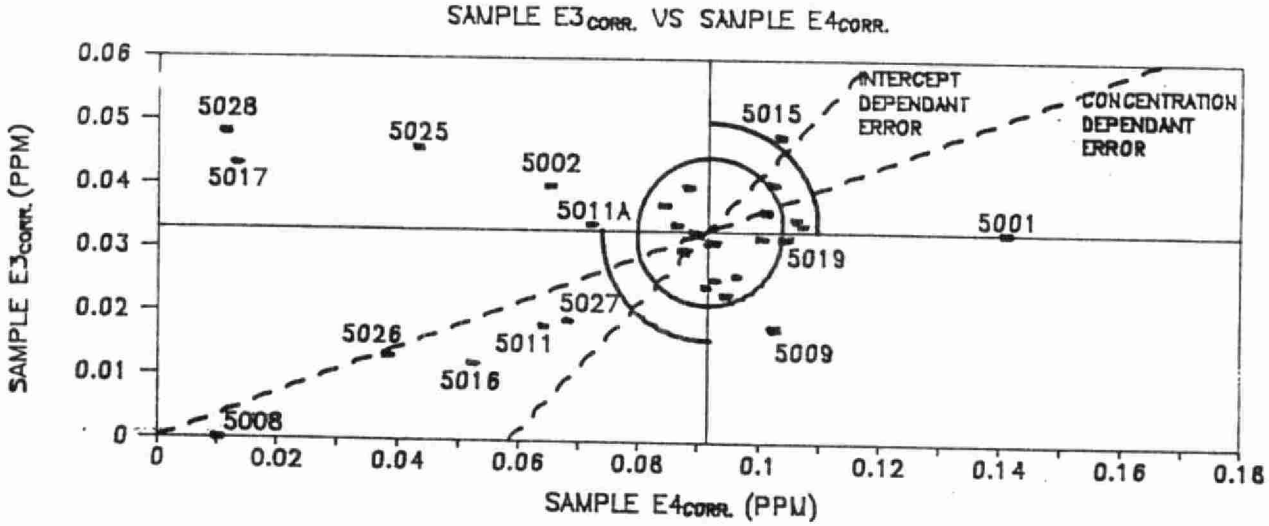
INTERLABORATORY STUDY 90-1: CYANIDE

MATRIX: REAGENT WATER



LABORATORIES 5008, 5021 & 5032 ARE OUTSIDE SCALE

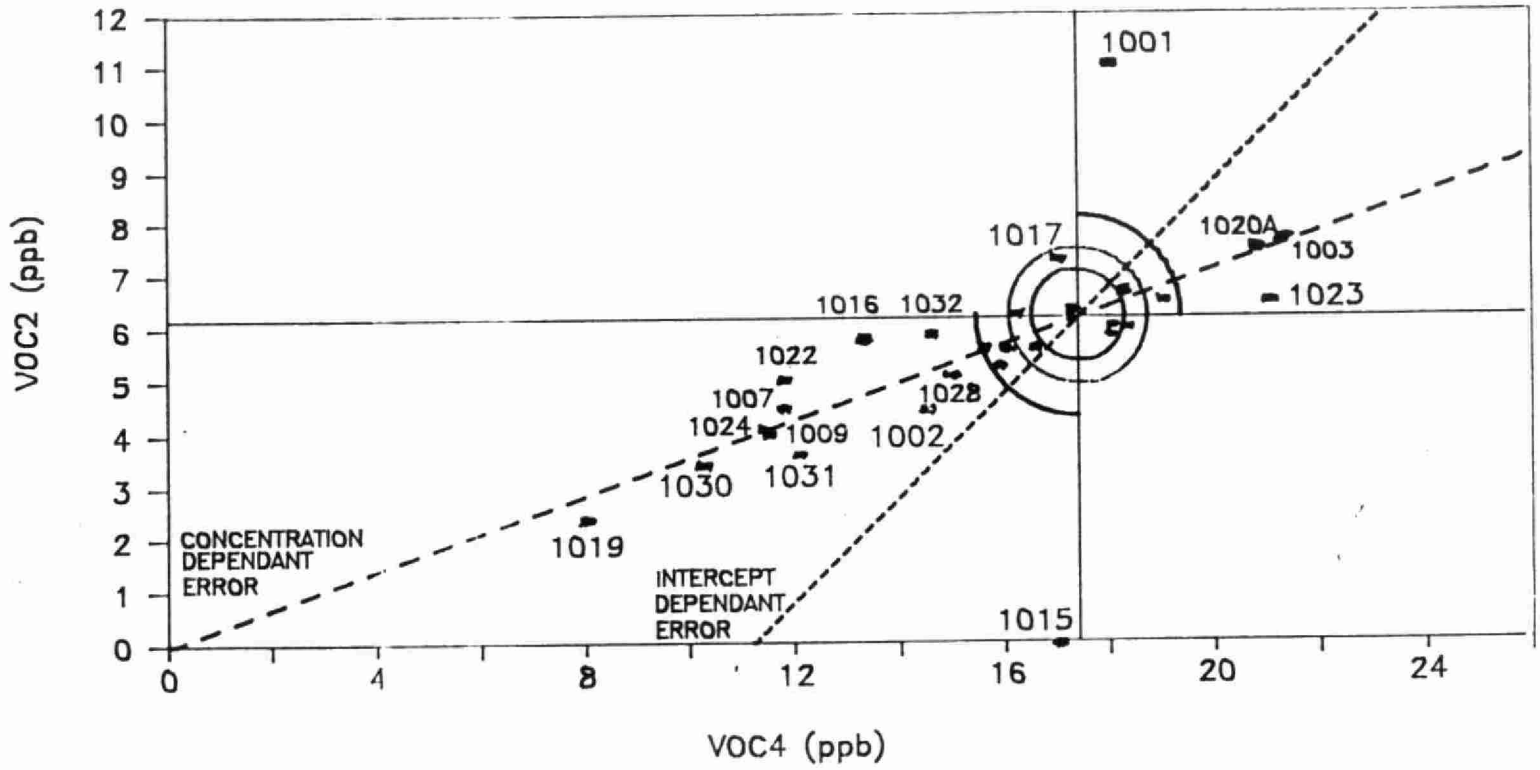
MATRIX: LAKEVIEW STP EFFLUENT



LABORATORIES 5021, 5023 & 5032 ARE OUTSIDE SCALE

INTERLABORATORY STUDY 90-5: BTX & ACRYLONITRILE

FIGURE 5: TOLUENE – VOC2 vs VOC4



ROUND ROBIN STUDIES

JUDITH SZEKELY

HAMILTON-WENTWORTH REGIONAL LABORATORY

Environmental labs today vary from small industrial or government owned facilities to sophisticated commercial or specialized governmental set ups. They all produce **numbers** which have an impact on industries, local economy and the community.

Problems facing environmental laboratories:

* Lack of a regulatory body who enforces:
  - GLP.
  - Universal methods.
  - Establish acceptable standards of instrumentation.
* Small facilities with not always qualified personnel and very limited resources.
  - Lack the possibility of extensive QA/QC.
  - Method development.

All these facilities with a large range of possibilities produce **data.**

For this data to have any meaning, in other words, to lend them consistency and a level of confidence need arises for a regulatory body. All these labs have to be brought to a common denominator.

We do not have that regulatory body at this time, and it seems to me that nobody really wants to assume this responsibility (not even, and especially not our gracious hosts, right here, the MOE). Different agencies cover part of the activities, such as CAEAL for certification, ACPO, licencing of the chemical professionals, etc.

We find under the present situation a solution to these problems in running **Round Robin** studies.

I guess we were not the only ones to discover that, so what happened - just like with any other commodity, round robins were set up by chemicals suppliers, CAEAL and others.

We are at the point now that it is difficult to choose the right study to partake in.

For a round robin study to be efficient it should satisfy the following requirements:

* Samples have to be certified, with all the QA/QC parameters defined in advance (detection limit, range, assay value, and method used to determine it).
* A set of samples should include:
  - Standards.
  - Test samples.
  - Spikes.

Data should be summarized and analyzed statistically to type of laboratory and level of instrumentation (manual or automated) available, otherwise results are misleading.

* Communication network - to coordinate and give advance notice of sample arrival to ensure laboratories are able to handle it.

* The "body" coordinating the study should have proven credibility.

Taking part in a round robin, might become fairly expensive and laborious, and quite a nuisance; since we are not quite regulated in this field why bother would many managers/owners ask.

Being part of such a study has great advantages:

* Raises confidence in the quality of the laboratories results, lends credibility to the facility.

* No matter how good the laboratories own QA/QC program can be, there might be a systematic error producing consistently erroneous results for a given parameter.

* Will provide valuable support data in court litigation.

At this point we arrived to MISA. Up to here, we outlined the QA/QC aspect of round robins in ensuring that quality results are supplied to the enforcement personnel.

There may be another aspect to it, though. MISA provides for the municipalities not only to monitor industries through their own tests, but also by accepting documentation provided by industries, as to their "good" performance.

The industries, on the other hand, can produce this documentation by using certified commercial labs or their own facilities. In this second case, these facilities should be asked to demonstrate the validity of their documentation:

* Industrial lab should be inspected for GLP, documents, QA/QC.

* Participation in round robin studies, and proof of quality of results should also enable industries to support their results.

ROUND ROBIN STUDIES

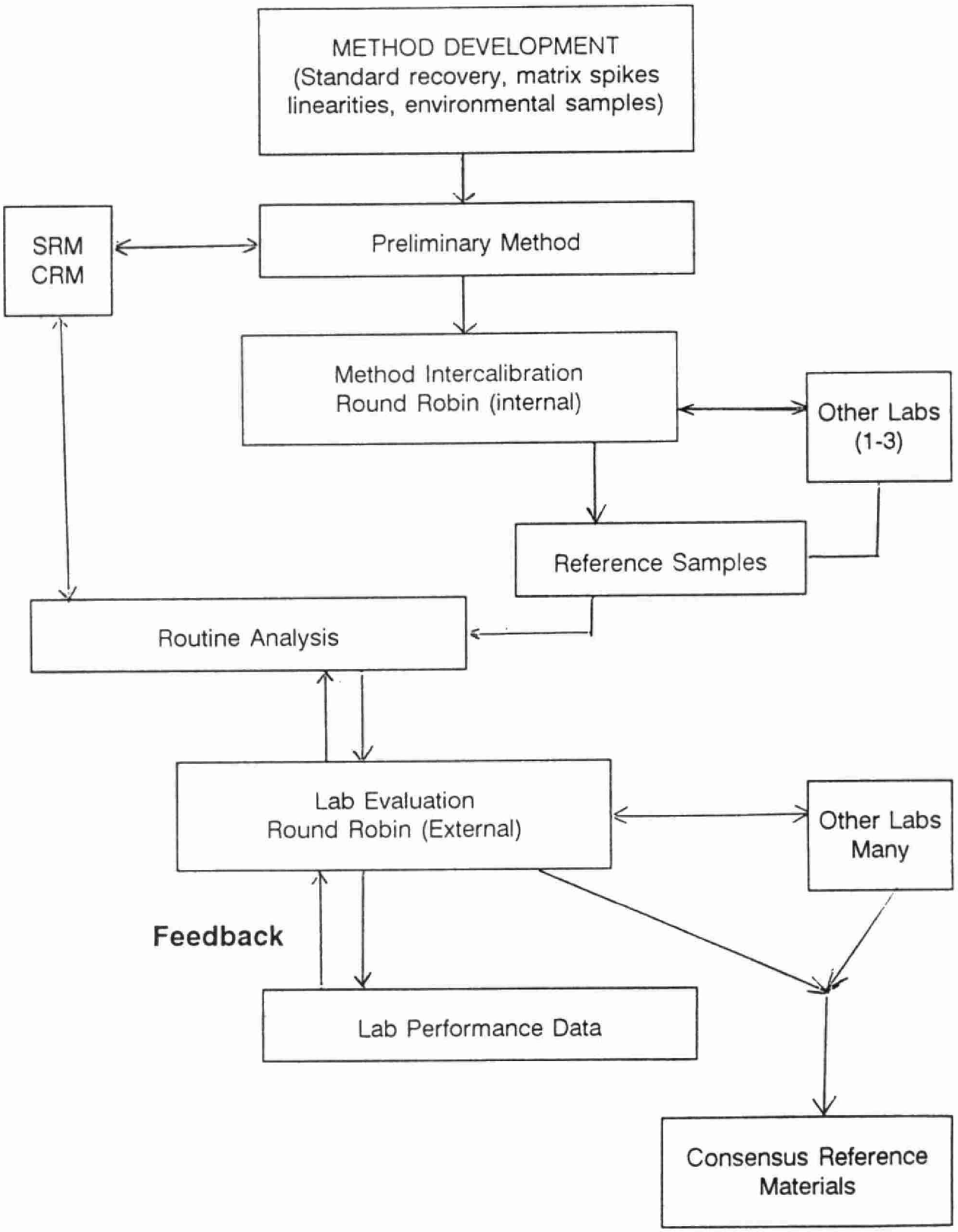
BRIAN FOWLER

AXYS ANALYTICAL LABORATORY

ROUND ROBIN

DEFINITION: A STUDY IN WHICH ONE OR MORE SAMPLES ARE ANALYSED INDEPENDENTLY BY SEVERAL ANALYSTS FOR THE SAME COMPOUNDS.

ROLE OF ROUND ROBINS IN THE EVOLUTION OF A METHOD



THE PROS AND CONS OF ROUND ROBIN EXERCISES FOR PRIVATE LABS

PROS — THE BENEFITS

1. Detect systematic method problems.
2. Detect errors in analytical standards.
3. Independent analysis can provide greater confidence in data.
4. A high ranking may benefit a lab and may serve as a promotion for the analyses involved.
5. Potentially could generate more Reference Materials.
6. The exercise can validate a developed method.

CONS — THE DISADVANTAGES

1. Round robin work may conflict with work load.
2. Added costs and commitment of staff and resources.
3. There is the possibility that a low ranking may work against a lab when not at the top.
4. Round Robins emphasise "consensus values" which may not be the 'design' or correct values due to common interferences or method differences.

PROBLEMS WITH ROUND ROBINS

---

1. No feedback report from submitted data.

2. No detectable analytes in the round robin samples.

3. Client-supplied surrogates contaminated with a target analyte and allowed no mechanism for blank correction.

4. Confidentiality not assured.

5. Data may not be evaluated in as an anonymous set.

6. Calculation methods may bias results especially for detection limits.

7. *Consensus value may not be 'correct'.

SUGGESTIONS FOR MORE USEFUL ROUND ROBINS

---

1. Round Robins are most useful to participating labs when there is timely feedback. A detailed discussion and comparison can come later.

2. The reporting requirements and evaluation criteria should be clearly laid out in documentation provided to participants.

3. Analyte levels in Round Robin samples should preferably not be at detection limits but at least ten times typical detection limits. Required detection limits should be stated.

4. Round Robins work well for single analytes but are less reliable for group totals. The problem of group totals may need to be evaluated using different criteria than for single component analytes.

5. When no SRM/CRM is available comparable with the round robin sample, sufficient sample should be prepared to allow it to be used as a "consensus" reference material.

6. Round robins should be scheduled for less busy periods. Perhaps a questionnaire could identify a preferred period.

7. Ranking participants is contentious; there will always be winners and losers when all may be qualified, (or conceivably none are qualified). Perhaps a better way may be to classify labs as "currently qualified" for a specific analysis.

ROUND ROBIN STUDIES

- DESIGN, IMPLEMENTATION AND VALUE

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QUOTE FROM A FAMOUS CHEMIST:

All you have to do to get the right answer is to send the sample to dozens of labs around the world, throw out the outliers, and take the median. That will be close to the truth.

Claude W. Sill
US Dept of Energy

WHAT IS A ROUND ROBIN

* Several identical sets of Perform Evaluation samples sent to a group of labs for analysis

* Results are analyzed, and a report prepared

* An experiment designed to achieve an objective

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WHAT CAN A ROUND ROBIN DO

* Provides useful information on variability in measurements within labs

* Identifies the sources of variability in datasets

* Provide feedback on performance to labs and to others

* Identifies weak laboratories and can provide clues to the source of the problem

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WHAT INFORMATION CAN BE PROVIDED

* Performance of different methods
    - specify methods exactly
    - bias, precision, ruggedness
    - methods failure hypotheses
    - interference assessment
    - assessment of useful range and scope
* Snapshot of Data Quality as it exists with existing methods
* Comparison of the instrument

WHAT INFORMATION CAN BE PROVIDED

* **Identify specific problem areas**
  - **calibration curves, series of standards**
  - **instrument (injection ready)**
  - **method**
  - **blanks, extrapolate to zero**
  - **matrix effects, series of sample spikes**

* **Comparison of groups of labs**
  - **Commercial vs Non - Commercial**
  - **Canada vs others**
  - **individual lab compared to the group**
  - practical multi lab MDL (PQL) assessment

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PQL

practical quantitation limit

EPA approach

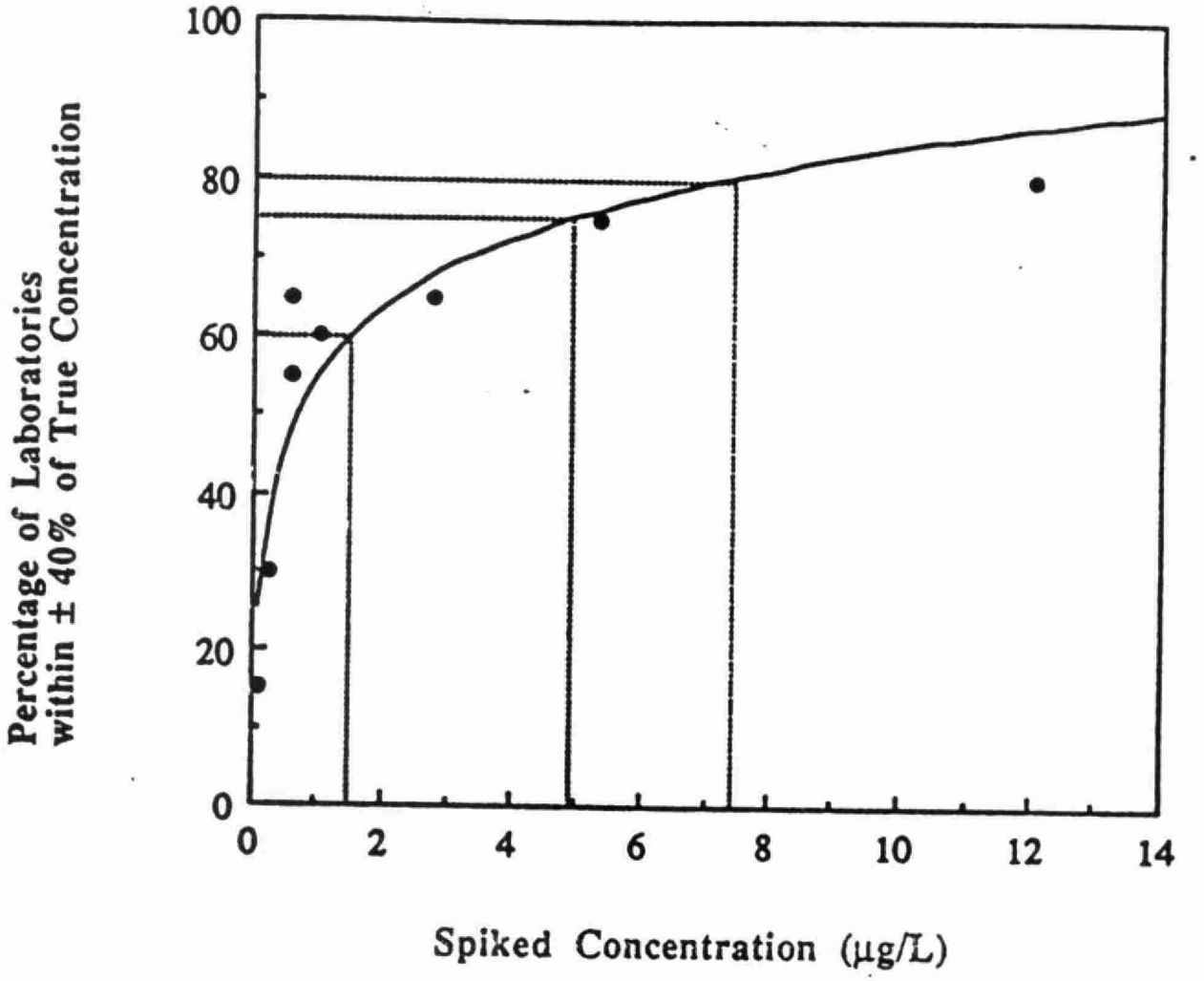
Concentration above which 80% of labs can get within 40% of true value

Useful for multi-lab datasets

Reflects differences(biases) due to

instrument, stds, analysts, methods

Figure 13 - Percentage of Laboratories within ± 40% of True Concentration for Benzene



DESIGN

* range
* high and low duplicate
* matrix (natural or synthetic)
* blank where applicable
* stability
* homogeneity
* interference
* contamination
* loss

HOW TO DO A ROUND ROBIN

* Identify labs that are willing to participate

* Prepare samples according to design
  - Usually of known composition
  - Standards, blank, real samples
  - Reference materials characterized by a number of labs
  - bottled in large runs
  - Homogeneity of all bottles
  - Stability by preservation & controlled temperature
  - Make sure there is no contamination or loss

* Ship the samples as soon as possible

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WASTEWATER TECHNOLOGY CENTRE

- is both a participant in, and a provider of round robin studies
- uses round robin data as part of internal QC activities
- uses round robin data to validate the samples used
- provides quick feedback to participants in WTC operated round robins
- has done AOX, SS, BOD, pH, P, CN

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TABLE 2
ANALYTICAL RESULTS

LAB	SAMPLE 1	2	3	4	5	6
1	259.0	7.3	3.9	40.0	8.1	4.0
2	213.0	6.1	3.0	33.5	7.7	5.0
* 3	679.0	620.0	2.1	236.0	9.6	3.7
4	257.0	7.3	1.8	40.0	8.0	4.2
* 5	209.0	13.8	11.3	42.5	31.3	16.3
6	———	———	———	———	———	———
7	202.0	5.7	2.6	29.4	7.1	3.7
8	194.0	8.8	3.6	35.6	6.8	5.3
9	229.0	5.9	3.3	33.1	6.5	2.9
10	247.0	6.2	3.5	37.0	8.5	3.7
11	251.0	7.2	3.8	38.4	7.8	3.8
12	208.0	6.1	3.1	34.4	7.1	3.6
13	203.0	4.6	2.4	30.5	7.4	4.1
14	186.0	6.7	3.1	30.0	7.1	3.4
15	214.0	5.9	2.9	37.3	7.3	3.7
16	220.0	5.2	2.4	30.0	6.9	3.3
17	209.0	4.5	2.7	30.1	6.3	2.7
18	209.0	5.1	2.3	29.8	7.5	3.5
19	198.0	3.7	1.8	28.1	6.9	3.6
20	204.0	5.3	2.8	29.0	7.6	4.0
21	207.0	5.1	2.7	31.1	7.7	4.0
22	212.0	5.3	2.7	28.2	7.8	4.0
23	245.0	6.1	3.2	38.0	8.8	3.8
24	170.0	5.2	2.8	25.0	7.0	3.3
25	250.0	4.8	1.9	34.0	8.0	3.4
26	286.0	5.0	2.7	35.0	7.4	3.9
MEAN	221.0	5.8	2.8	32.9	7.4	3.8
MEDIAN	212.0	5.7	2.8	33.1	7.4	3.7
STDEV	27.7	1.1	0.6	4.2	0.6	0.6
C.O.V	12.6	19.5	20.6	12.7	8.2	15.1
MIN	170.0	3.7	1.8	25.0	6.3	2.7
MAX	286.0	8.8	3.9	40.0	8.8	5.3

* DATA NOT USED IN STATISTICAL CALCULATIONS

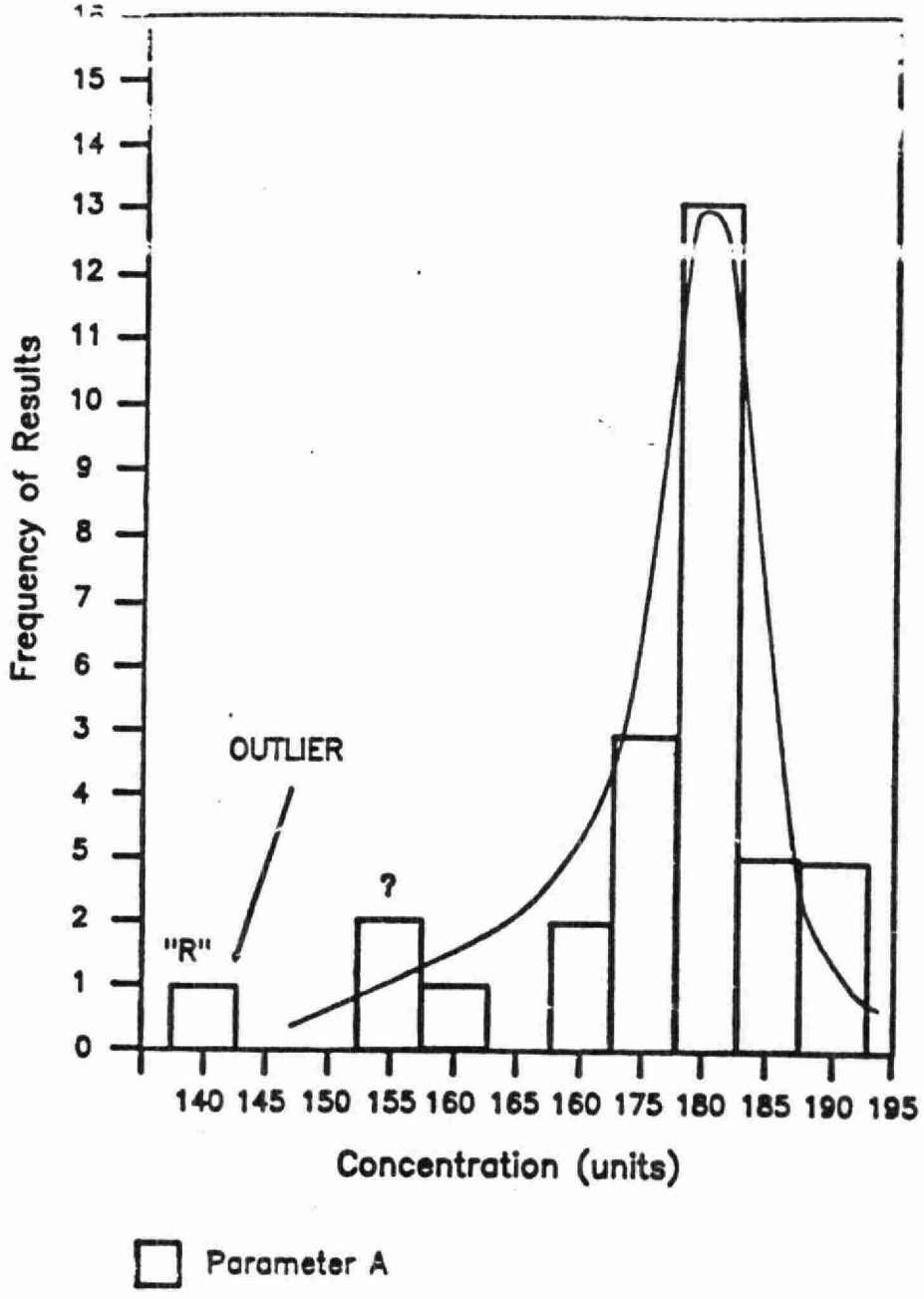


Fig. 12 An Example of a Statistical Outlier (Grubbs)

Data Evaluation for FP & PPWB QA Programs

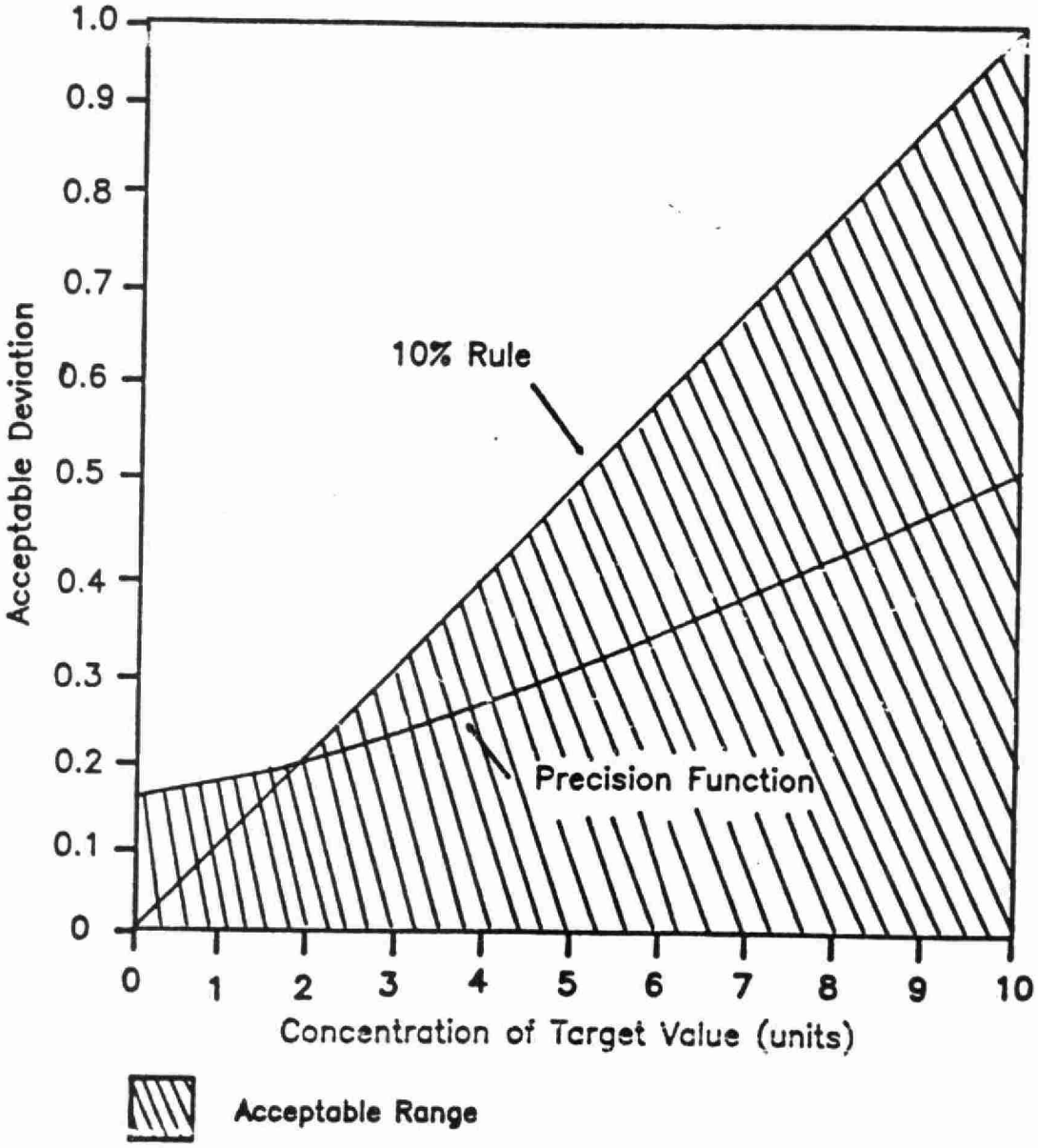
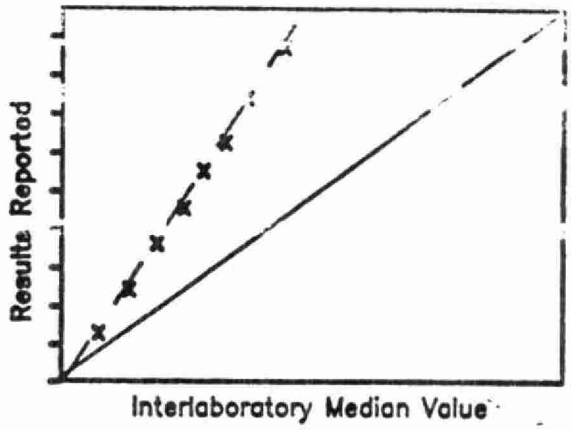


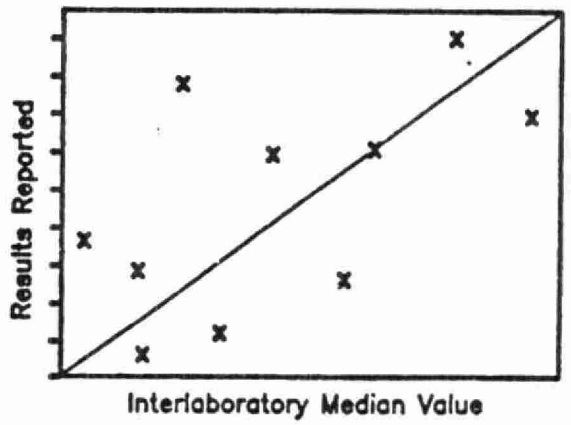
Fig. 11 Evaluation of Data based on 10% & 1 Std. Dev. Rule

CASE A



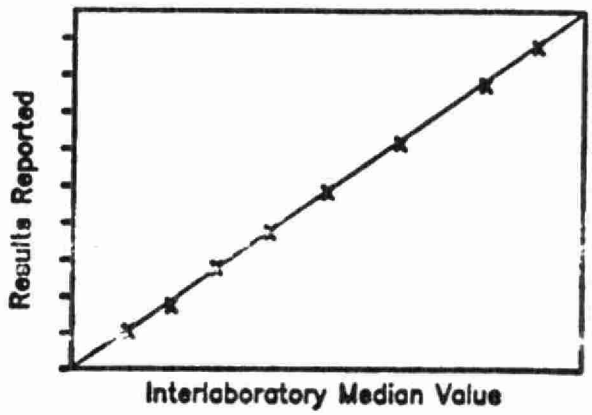
Severe high bias
(inaccurate)

CASE B



Very erratic
(very imprecise)

CASE C



Satisfactory
(very good)

Fig. 2a Some typical types of Laboratory Performance revealed by External QA Studies

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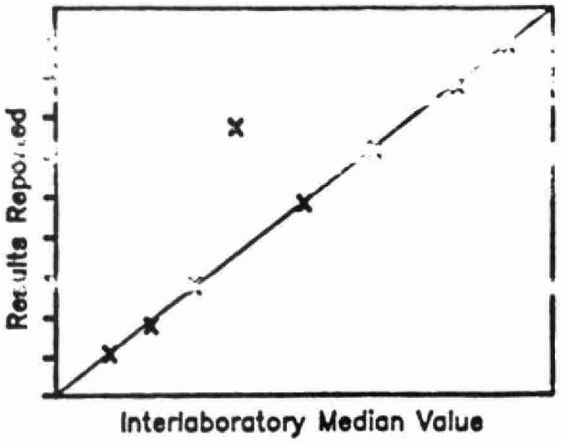
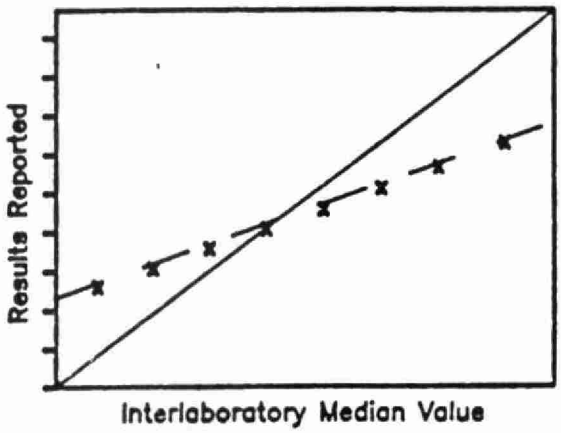
CASE D



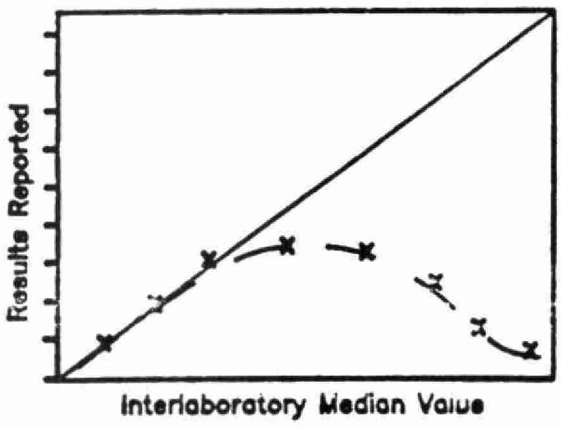
Out of Control

CASE E



Biased with
Blank Problem

CASE F



Methods Failure

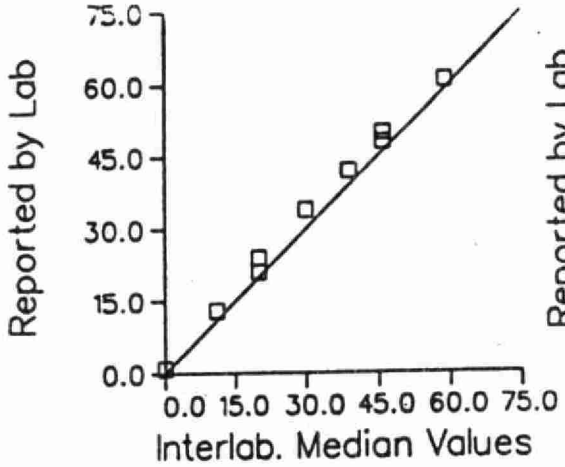
Fig. 2b Some typical types of Laboratory Performance revealed by External QA Studies

wtc

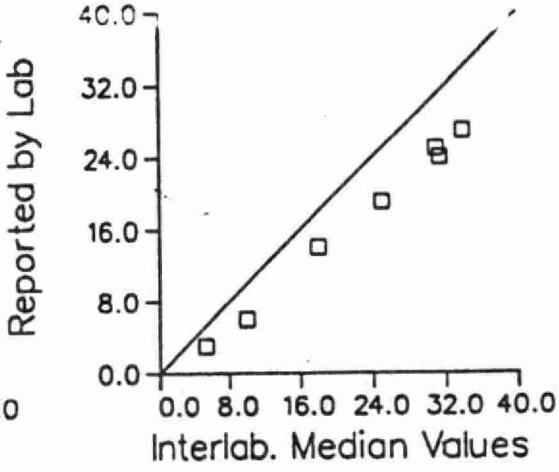
Laboratory: W0072

Comparison of Results Reported versus
the Interlaboratory Median values

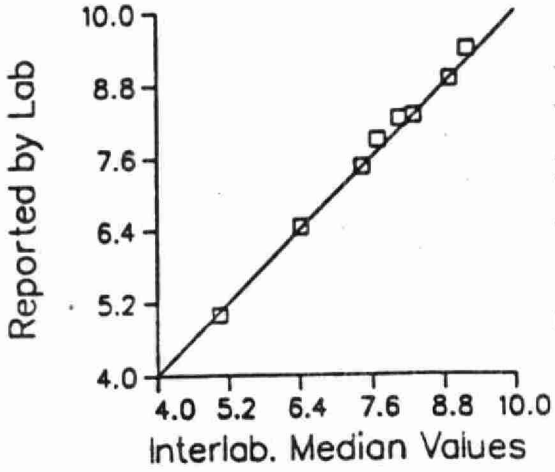
SS ,mg/L



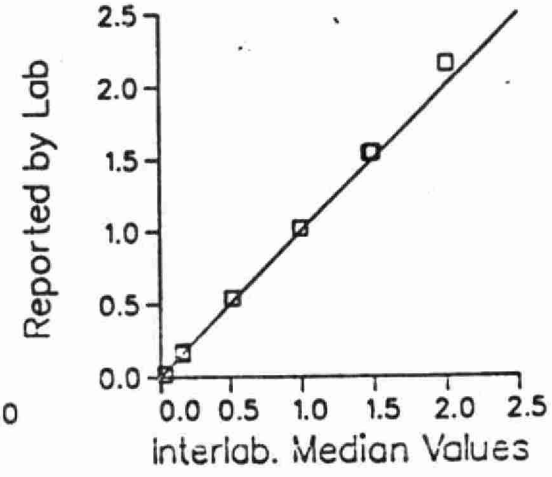
BOD ,mg/L



pH ,pH UNITS



Total P ,mg/L

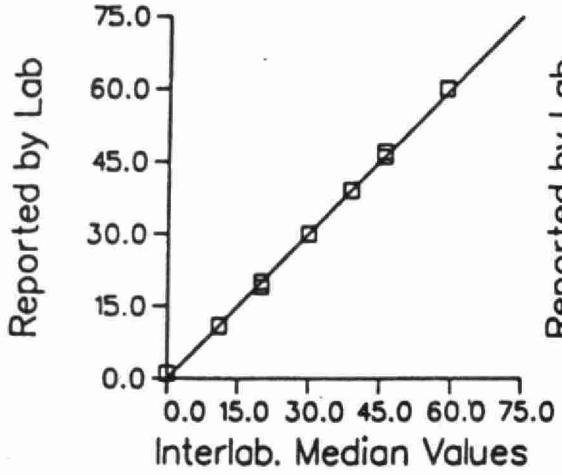


Lab Code: W0072

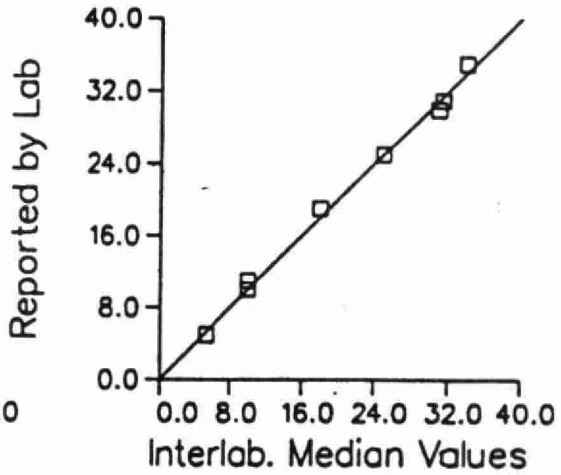
Laboratory: W0506

Comparison of Results Reported versus the Interlaboratory Median values

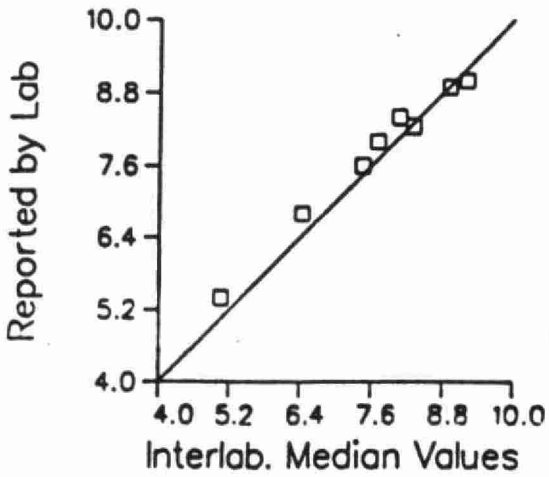
SS ,mg/L



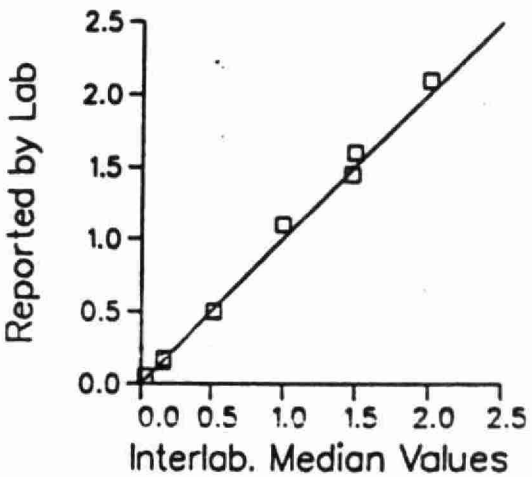
BOD ,mg/L



pH ,pH UNITS



Total P ,mg/L



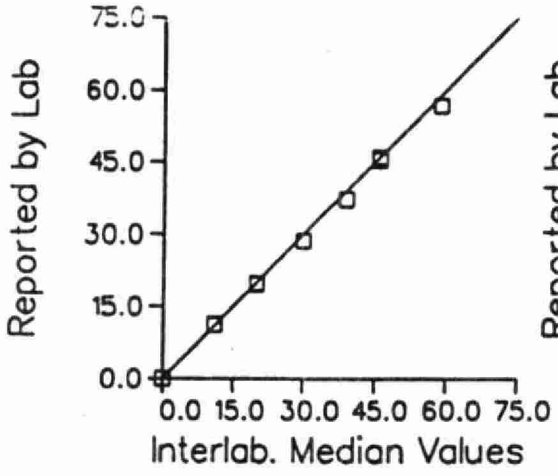
Lab Code: W0506

wtc

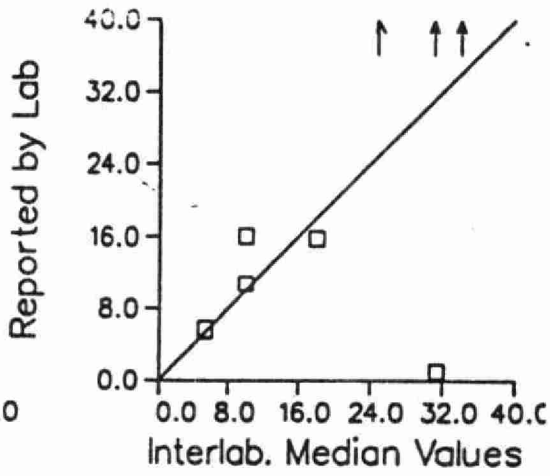
Laboratory: W0489

Comparison of Results Reported versus
the Interlaboratory Median values

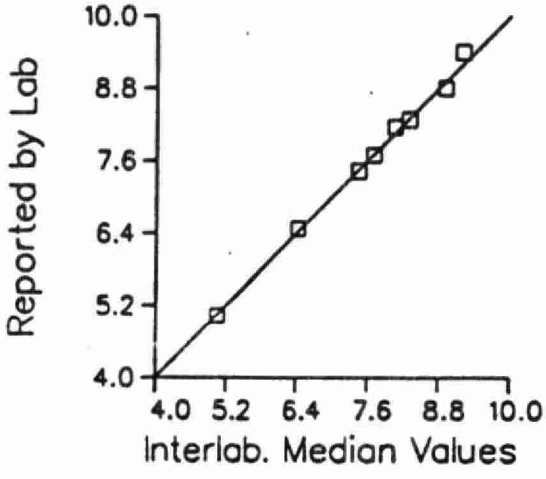
SS ,mg/L



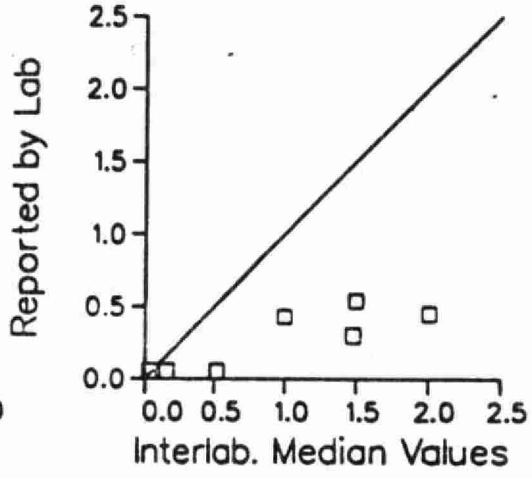
BOD ,mg/L



pH ,pH UNITS



Total P ,mg/L

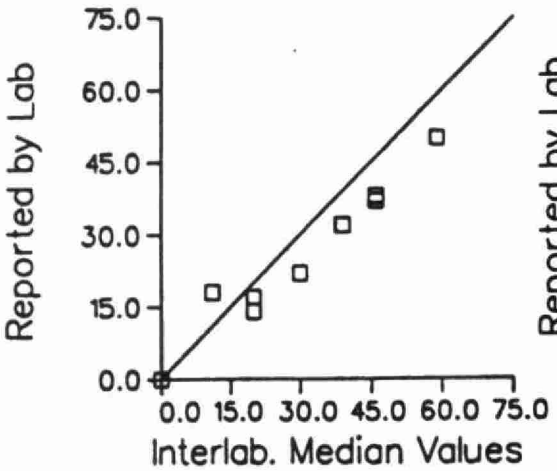


Lab Code: W0489

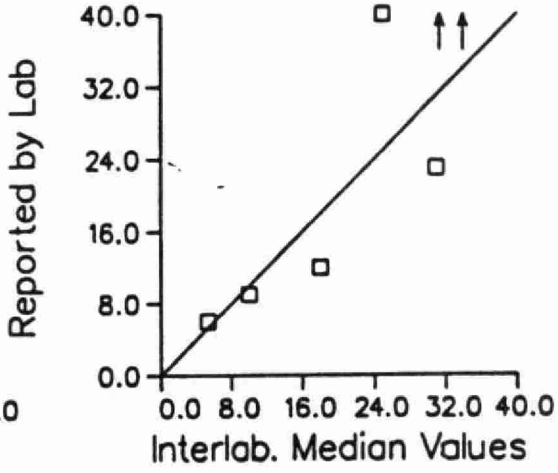
Laboratory: W0404

Comparison of Results Reported versus
the Interlaboratory Median values

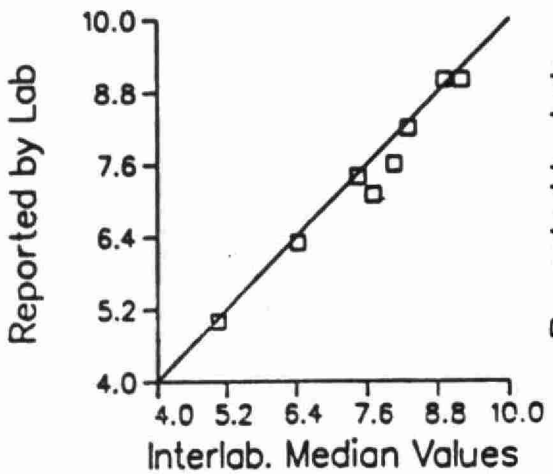
SS ,mg/L



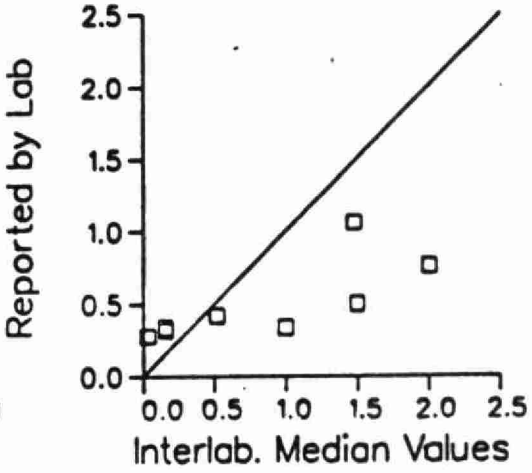
BOD ,mg/L



pH ,pH UNITS



Total P ,mg/L

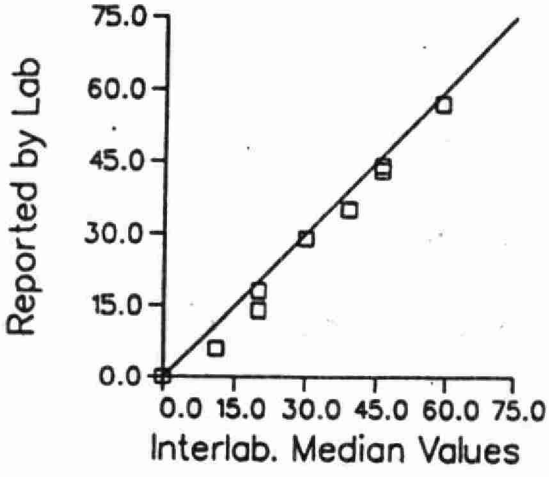


Lab Code: W0404

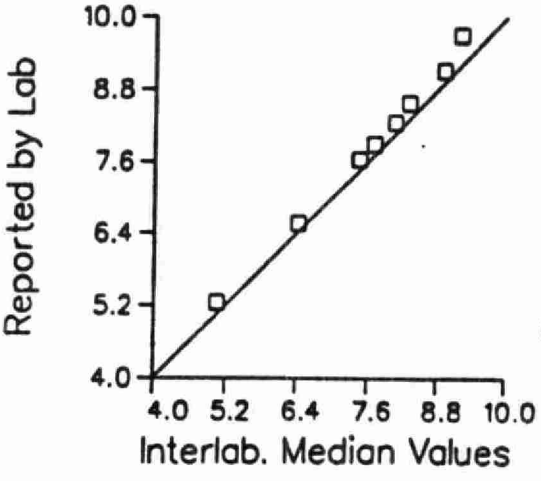
Laboratory: W0198

Comparison of Results Reported versus the Interlaboratory Median values

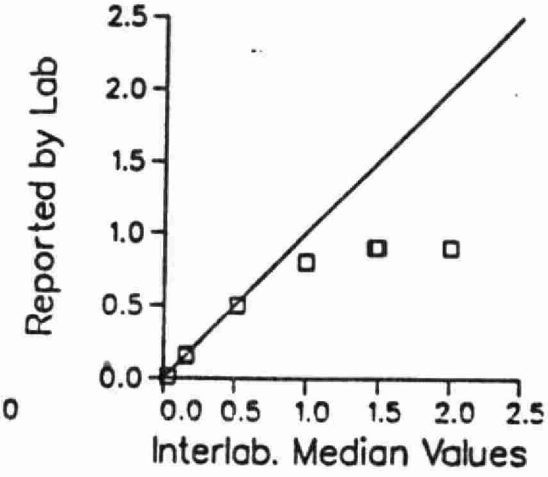
SS ,mg/L



pH ,pH UNITS



Total P ,mg/L



Lab Code: W0198

wtc

WHAT USE IS A ROUND ROBIN

USES FOR PARTICIPANTS:

- External QC, compare to peers
- feedback on data quality produced
- can identify and diagnose problems before your clients identify them for you
- due diligence in your QC efforts
- evaluate your lab methods
- useful info to educate clients

USES FOR PROVIDERS OF ROUND ROBINS:

- Info on performance of the lab community
- Info on specific labs
  - usually confidential
  - useful for contracting analysis
- Info on expected quality of a multilab database
- Info on method performance
- Data on "True Value" of a reference material by a selection of methods
- Get really good methods specifications on your own lab

wtc

USES FOR CONSUMERS OF DATA:

- individual lab performance
    - need to know basis
- expected database quality
- Can realistically identify DQO's and project plans
- Can identify trends in data quality
- Can force improvements in data quality

wtc

USES FOR REGULATORS AND DECISION MAKERS:

- To ensure that decisions are made on data of known quality and that the limitations and variability in the data are taken into account

ANALYTICAL CHEMISTRY UNDER REGULATIONS

OTTO HERRMANN
J.A.W.G.

INDUSTRY	MOE
"IDEAL" METHOD	LIMITED TIME & RESOURCES
+	+
EXTERNAL LIMITATION (SPECIFIC METHOD & VARIATION)	POLITICAL INTERFERENCE VS TECHNICAL JUDGEMENT
+	+
LEGALLY RESPONSIBLE	MULTIPLE INTERPRETATIONS OF "UNAMBIGUOUS" DESCRIPTION
+	
INTERPRETATION (WRITTEN BY LAWYERS)	
+	
TIME LIMIT	

COMMON GOALS

BUT
LOCAL STRESSES ====>

POLARIZED VIEWS

Joint Analytical Working Group

- INDUSTRIAL SECTORS
  - Inorganic Chemical
  - Iron & Steel
  - Metal Casting
  - Mining & Industrial Minerals
  - Organic Chemical
  - Petroleum Refining
  - Power Generation
  - Pulp & Paper
- Environment Canada (Wastewater Technology Centre)
- Ministry of the Environment
- "VISITORS"

PURPOSE: DEAL WITH ANALYTICAL ISSUES

GENERALLY TECHNICAL REPS VS ADMINISTRATORS

WIDE REPRESENTATION BECAUSE ISSUES CUT ACROSS SECTORS

PROVIDES "ONE-STOP SHOPPING" ON ANALYTICAL ISSUES

"VISITORS" TO DISCUSS SPECIAL CONCERNS OR TO PROVIDE EXPERTISE FROM BOTH MINISTRY AND INDUSTRY

SO WHAT'S HAPPENED?

- FORMATION PROPOSED BY DON KERR (CONSULTANT) AUGUST 1990
- 1ST MEETING HELD OCTOBER 1990
- 12TH MEETING WILL BE IN MAY 1992.

A MEETING IS NOT AN "EVENT" IN ITSELF

WHAT'S BEEN DISCUSSED?

- 1. REHASH THE ANALYTICAL AND SAMPLING PROTOCOL DOCUMENT
- 2. QA/QC - REQUIREMENTS, CHARTING, OUT OF CONTROL
- 3. LMDL - HOW AND WHEN
- 4. NIMMP (NEW INSTRUMENTAL MEASUREMENT METHOD PRINCIPLES)
- 5. RECORD KEEPING AND DATA RETENTION
- 6. MIDES AND DATA ENTRY - PROBLEMS AND REVISIONS
- 7. LABORATORY WORKSHOPS - THIS IS IT!
- 8. HEXACHLOROCYCLOPENTADIENE (IN ATG23) - FORGET IT
- 9. PHENOLIC CONTAMINATION IN AUTOSAMPLERS - WATCH IT
- 10. PHENOLIC PRESERVATION - ATG14 (4AAP PHENOLICS) & ATG20
- 11. DIOXINS
- 12. ION CHROMATOGRAPHY FOR FLUORIDE AND SULFATE - SURE, WHY NOT?
- 13. SOLVENT EXTRACTABLES (ATG25; OIL & GREASE))
- 14. VCM/BTX (VINYL CHLORIDE MONOMER, BENZENE, TOLUENE, XYLENE)
- 15. TOTAL PHOSPHORUS AND STANNOUS CHLORIDE - DON'T DO IT!
- 16. ANTIMONY AND FALSE POSITIVES - CAN'T PRECHARGE PET BOTTLES
- 17. ALKYL LEADS (ATG13)- GRAB SAMPLING ONLY
- 18. STYRENE AND O-XYLENE - CHROMATOGRAPHIC RESOLUTION RESOLVES THE PROBLEM

PRESERVATION OF PHENOLICS

- HOW IT WAS (IS):

  SULFURIC ACID TO pH 1.5 - 2 : 10 DAYS
  PHOSPHORIC ACID / COPPER SULPHATE PRECHARGED: 4 DAYS

- MULTI-LAB STUDY (MINISTRY PLUS INDUSTRY)

  1. STANDARDS IN DEIONIZED WATER + PRESERVATIVES => OK 30+ DAYS

  2. REAL SAMPLES "AS IS", SPIKED SAMPLES + PRESERVATIVES
     STUDY JUST FINISHING
     LOOKING HOPEFUL

- BUT

  WHAT ABOUT ATG20? NO PRESERVATIVES FOR 30 DAYS.

SOLVENT EXTRACTABLES (ATG25)

- GRAB VS AUTOSAMPLER - OBVIOUS PHASE SEPARATION?
- COMMON METHODS USE:
  - CHLOROFLUOROCARBON (FREON 113)
  - DICHLOROMETHANE (DCM)
- BUT:
  - EXTRACTION EFFICIENCIES NOT SAME - WHAT WILL BE REGULATED?
  - CFC USE BANNED IN 1994
  - MUCH HISTORICAL DATA BASED ON FREON
  - MUNICIPALITIES SPECIFY FREON
  - EPA CONSIDERED HEXANE / METHYL TRIBUTYL ETHER (20/80)
    DORMANT
  - EUROPE JUST ABANDONED CCL4 FOR FREON
- INTERIM:
  - TENDING TOWARD DICHLOROMETHANE
  - RECOMMEND NOT REGULATE BASED ON FREON DATA
  - POSSIBLE MONITORING PHASE BEFORE SET REGULATORY LIMIT
- INVESTIGATION OF SOLID PHASE EXTRACTION ALTERNATIVE
- UNRESOLVED: WHY? WHAT IS THE PURPOSE?

DATA ENTRY & MIDES

- PROBLEMS:DIFFICULT ENTRY
  - ORIENTATION TO SMALL MANUAL OPERATION
  - REVISIONS DESTROY BUG-FIXES
  - DIFFICULT COMMUNICATION

- WHY JAWG AS THE INTERFACE?

  TECHNICAL PROBLEM
  COMMON TO ALL
  SINGLE INTERFACE BETWEEN MINISTRY AND INDUSTRY

- WHERE ARE WE?

SUMMARY & THE FUTURE

- THE PROCESS WORKS
- INCORPORATE MUNICIPAL SECTOR - IN PROCESS
- COMMERCIAL LABS - BEGIN PROCESS OF REPRESENTATION
- NEEDED MINISTRY IMPROVEMENTS

  PRESENT UNIFORM, EASY-TO-ACCESS FRONT
  BETTER PROCESS FOR ACTION ONCE CONSENSUS REACHED
  SORT OUT THE INTERNAL BATTLES FOR POWER

- QUOTABLE QUOTES

  "IF THE REGULATIONS DON'T FORBID IT, DO IT."
  "IT'S EASIER TO GET FORGIVENESS THAN PERMISSION"
  "IF THE EDICTS COME OUT WITH ONE LABEL, SO SHOULD THE SOLUTIONS"

SECTION FOUR

DAY 2 - SESSION THREE PRESENTATIONS

CHAIRPERSONS -	G. STEINKE, MOE R. CLEMENT, MOE
PROTOCOL DOCUMENT -	P. BAULU, MOE
NON-COMPLIANCE/ENFORCEMENT -	C. DOEHLER, MOE
DETECTION/LOW LEVEL DATA -	D. KING, MOE
CAEAL -	B. TRAVERSY, CAEAL G. CRAWFORD, MOE

PROTOCOL FOR THE SAMPLING AND ANALYSIS OF INDUSTRIAL/MUNICIPAL WASTEWATER

SAMPLING/ANALYTICAL PROTOCOL

Introduction

- PROTOCOL WILL BE REFERENCED IN LIMITS REGULATIONS
- WILL BE CONSIDERED AS A LEGAL DOCUMENT
- REQUIREMENTS FOR SAMPLING AND ANALYSIS OF EFFLUENTS UNDER LIMITS REGULATIONS
- EASE OF REVISION/AMENDMENT
- MAY BE REFERENCED IN OTHER PROGRAMMES (SUCH AS CERTIFICATES OF APPROVAL)

SAMPLING/ANALYTICAL PROTOCOL

Introduction

- REGULATION REQUIREMENTS EXPRESSED IN TECHNICAL/SCIENTIFIC LANGUAGE
- RECOMMENDATIONS & SUGGESTIONS INCLUDED
- ADDITIONAL TEXT FOR CLARIFICATION OF MOE INTENTIONS & EXPECTATIONS
- FOR EASY REFERENCE, ATG GUIDE LISTS ALL REQUIREMENTS FOR THAT GROUP OF PARAMETERS

SAMPLING/ANALYTICAL PROTOCOL

Introduction

OUTLINES PRINCIPLES TO BE FOLLOWED FOR:

- SAMPLING
- PRESERVATION
- STORAGE
- ANALYSIS
- QUALITY CONTROL

SEPARATE DOCUMENT OUTLINES DATA REPORTING PROTOCOL

SAMPLING/ANALYTICAL PROTOCOL

Data reporting

- DATA PROTOCOL LISTS MINIMUM REQUIREMENTS FOR COMPLETE DATA BASE
- REPORTING REQUIREMENTS ARE EXPECTED TO BE STATED IN THE REGULATIONS
- REPORTING REQUIREMENTS MAY BE MINIMAL WIT BULK OF DATA RETAINED BY DISCHARGER FOR MOE REVIEW UPON REQUEST OR SUBMITTED TO MOE AT SPECIFIED FREQUENCY QUARTERLY ?)

SAMPLING/ANALYTICAL PROTOCOL

ATG guide

- SECTION 5, ATG GUIDE, SET-UP FOR QUICK & EASY REFERENCE
- TABLE FOR EACH ANALYTICAL TEST GROUP (ATG)
- EACH TABLE CONTAINS ALL INFORMATION FOR SAMPLING, ANALYSIS AND QC FOR THAT TEST

SAMPLING/ANALYTICAL PROTOCOL

Sampling

REQUIREMENTS LISTED FOR:

- SAMPLING TECHNIQUES
- SAMPLE CONTAINERS
- CONTAINER PRE-TREATMENT
- PRESERVATION
- STORAGE
- RECOMMENDED SAMPLE VOLUME
- PRECAUTIONS & REMARKS
- USER'S CHECKLIST IN SCHEDULE 2

SAMPLING/ANALYTICAL PROTOCOL
Analysis

OUTLINES ANALYTICAL PRINCIPLES FOR:

- SAMPLE PREPARATION & PRE-TREATMENT
- ANALYTICAL TECHNIQUE
- INSTRUMENTAL MEASUREMENT METHODS
- REPORTING UNITS
- PERFORMANCE CRITERIA/RMDL
- ALTERNATE & NOT RECOMMENDED METHODS ALSO LISTED WHERE APPLICABLE

SAMPLING/ANALYTICAL PROTOCOL
Quality control

REQUIREMENTS FOR TYPE & FREQUENCY OF LAB & FIELD QC SAMPLES:

LAB QC SAMPLES INCLUDE: METHOD BLANKS, SPIKED BLANKS, SPIKED SAMPLES & REPLICATES

RECOMMENDED FIELD QC SAMPLES: TRAVELLING BLANKS, TRAVELLING SPIKES & DUPLICATES

Presenting

Potential Non-Compliances/ Enforcement

Cathy Doehler
Laboratory Services Branch
Ontario Ministry of Environment

Non-Compliances/Enforcement

- inspection types/personnel
- lab compliance items
- report preparation
- follow-up action/personnel
- MOE compliance policy
- LSB services/lab responsibilities

MOE Inspection Personnel

- Regional staff
  (MISA and/or Abatement)
- Water Resources Branch
  (MISA Industrial, toxicity, flow monitoring)
- Laboratory Services Branch
- Investigations and Enforcement Branch

Inspection Types/Personnel

- spot
  (regional staff)
- routine
  (regional staff)
- detailed
  (region, LSB, WRB)

Inspection Areas

- site
- flow
- sampling
- analysis
- toxicity testing
- data reporting

Laboratory Inspection Areas

- sampling/preservation
- sample storage/transport
- sample analysis
- QA and control limits
- data reporting and interpretation
- staffing and safety

LSB INSPECTION PROGRAM

- 76 industrial labs
  25 commercial labs

- inspections prioritized
  -number of parameters analyzed
  -method complexity
  -location

Laboratory Compliance Items

- use of SRMs
- bench QC
- demonstration of control status
- mdl calculations
- calibration curves
- temperature maintenance

FUTURE LSB INSPECTIONS

- emphasize compliance items
- data consistency/improvement focus
- final details in development

GLP Suggestions

- sampling/
  preservation recommendations
- balance weight checks
- record keeping improvements
- control charting suggestions
- housekeeping improvements

COMPLIANCE

"a state achieved by adhering to the legislative and regulatory requirements of the Ontario Ministry of Environment"

- MOE compliance policy

Inspection Results

- prepare reports
  -commercial lab/LSB
  -regional inspection

- follow up action

LSB *will continue to:*

- perform lab inspections
- prepare inspection summary reports
- consult/advise regions/industry/labs/WRB on MISA-related laboratory concerns
- provide LSB MISA methods (BBS available)
- organize and manage interlab studies

LSB *will not:*

- approve/certify laboratories
- recommend commercial labs
- approve use of non-regulation methods
- provide standards or check samples
- lay or proceed with charges

MISA

"virtual elimination

of toxic and persistent

contaminants"

LOW-LEVEL DATA ISSUES

MISA PROGRAM

MISA WORKSHOP 1992

Don King
Environment Ontario
Laboratory Services Branch

MISA WORKSHOP 1992

LOW-LEVEL DATA ISSUES

Don King

DISCUSSION

This presentation shows diagrammatically the relationships among: performance criteria, performance indicators, low-level measurement practices, and data reporting practices. It also reviews the process for setting compliance limits based on MISA effluent data including the Long-Term Average (LTA) and the Variability Factor (Vf). It discusses the effect of low-level data censoring on the Limit Setting process. It also discusses the interpretation of data that falls above the compliance limit.

Fig.1 (left side)

For the MISA Monitoring Regulation laboratories were required to demonstrate their ability to measure at low-levels. For this purpose the regulation specified an upper limit for the laboratory's method detection limit (MDL) and required all laboratories to estimate their MDL using a specified protocol [1] since revised to address multi-analyte scan situations. For the purposes of this discussion the criteria value is RMDL, and the individual laboratory estimate is LMDL. In order to estimate LMDL the lab must calculate the within-batch standard deviation (Sw) for eight replicate analyses of a low-level spiked blank and multiply this value by 2.998.

Fig.1 (middle)

To ensure a reliable estimate of Sw, the lab is essentially required to be able to read results to the nearest RMDL/10. For MISA this value is labelled as W. It represents both a reporting increment (e.g when RMDL = 20 then results should be reported in steps not larger than 2, thus 18, 20, 22, 24 ...), and a lowest reportable value. When a measured result is below W, then it would be reported as 2 <W. The remark code <W signifies essentially a null response. For many labs the MISA W value falls between Sw/3 and Sw. Two other decision points were recognized. The code DL is set at the LMDL value and results below DL were coded <DL. This remark signifies that the presence of analyte in the sample is not entirely demonstrated. T is the code for the RMDL value, it represents 10 times W. Results below RMDL but at or above DL would be coded <T. This signifies a 'tentative' result.

Note: While a result coded <DL can be obtained even if the analyte is not present in the sample, the sample may still contain analyte at levels up to the reported value plus LMDL.

Note: Inability to measure a specific sample because of matrix effects required the lab to indicate an upper limit for the possible analyte level. Such data would be coded <.

Fig.1 (right)

For all sectors except Petroleum, laboratories were required to report results down to their LMDL value. They were permitted to report lower-level data. During the effluent data review, all results below RMDL/10 were equated to RMDL/10. The availability of this low-level data assisted greatly in the QA review of the effluent data. It provided better comparison between low-level effluent values and the corresponding travelling blank data, with the result that some parameters were 'deselected' as candidates for limit-setting. (Real data is easier to interpret than 'less than' data.)

Fig.2 (top)

Tentative effluent limits are estimated based on the long-term average (LTA) and variability (Vf) for the effluent data. The concentration limit is defined as LTA times Vf where Vf is the 99% confidence factor used to identify the point beyond which a result is taken as strong evidence that the actual concentration today exceeds the LTA value. For MISA, limits will be specified for loadings (concentration times flow). If a <T, <DL, or <W result is used to estimate loading, then the loading estimate should include a similar remark. For those parameters subject to effluent loading limits such data might not be irrelevant, since very high flows may combine with very low concentration estimates to cause an apparent exceedence.

Fig.3

Even though the Ministry may not that the company has failed to maintain an acceptable effluent quality until the 99% confidence value has been exceeded, the company should recognize that effluent quality might be a concern whenever the LTA value is exceeded. Thus if the value exceeds LTA + (Vf-1)/3 the confidence level is already approaching 80% that effluent quality is degraded.

Note that the Vf value will generally be much larger than the corresponding LMDL. The limits are based on a Vf derived from single estimates. It includes all sources of laboratory, sampling, and industry process variability. It may be best to perform only one analysis, reserving the option to repeat the measurement for only those cases where the measurement process is itself suspect. This includes evidence of poor recovery, poor blank estimates, etc., without reference to the impact of the result on the client. If laboratories perform replicate analyses whenever there is a risk that their client's effluent might be deemed out-of-compliance, care may be required in reporting the data. Thus should the lab report the highest, the lowest, the middle, or all values? A single estimate may be the safest approach?

Fig.4

Initially there was concern that inclusion of low-level data would lead to low estimates of LTA which would tighten the effluent quality criteria. It is now recognized that even though the LTA may be lower, the Vf value is generally much larger, so that the 'limit' is higher than if data is censored. In addition, the inclusion of low data often brings the LTA below the RMDL value. As a general principle such data is not considered sufficiently reliable to permit calculation of defensible limits. As noted earlier, such data was often similar to the travelling blank estimates and therefore led to deselection of the parameter for limit setting purposes.

Summary

The designation of an RMDL value ensured that all labs were on a level playing ground in terms of measurement capability. Actual sample matrix effects may forestall the analysis of the sample. And often matrix effects may cloud the interpretation of measurement data. But the availability of low level data has had a distinct positive effect on the evaluation of effluent data quality, and on the use of this data for possible limit setting purposes.

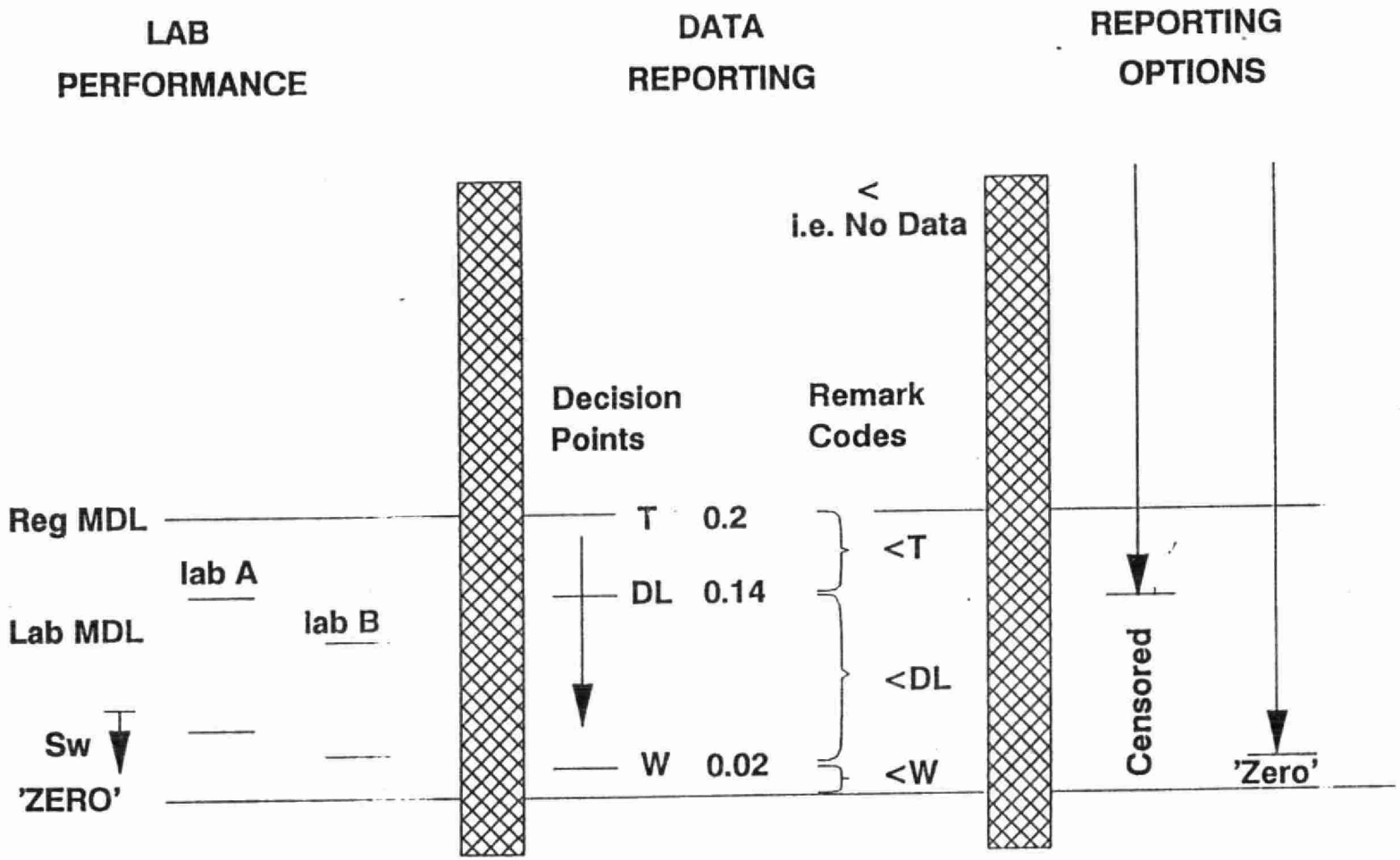
---

1) Estimation of Analytical Method Detection Limits, ISBN 0-7729-8817-x, (rev. June 1991) MISA document available from Communications Branch, MISA Office, or Lab Services Branch

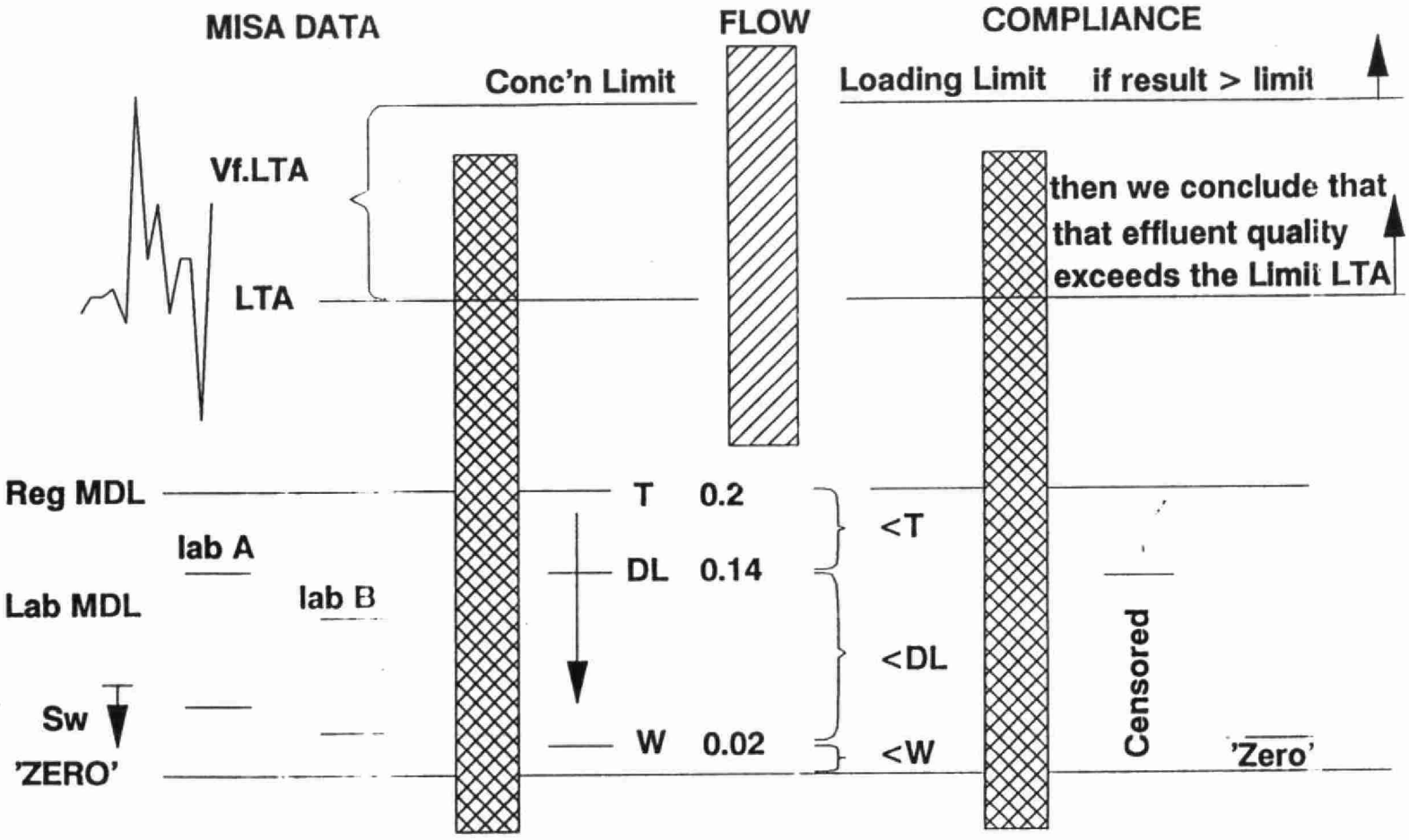
LOW-LEVEL DATA ISSUES: MISA

1. Adequate Sensitivity Regulation MDL
2. Demonstrated Ability Laboratory MDL
3. Consistent Reporting Increment W
4. Specific Decision Points DL T
5. Low-level Data Qualifiers <T, <DL, <W
   Inability to measure <, (<WE)
6. Censored and Non-Censored Data

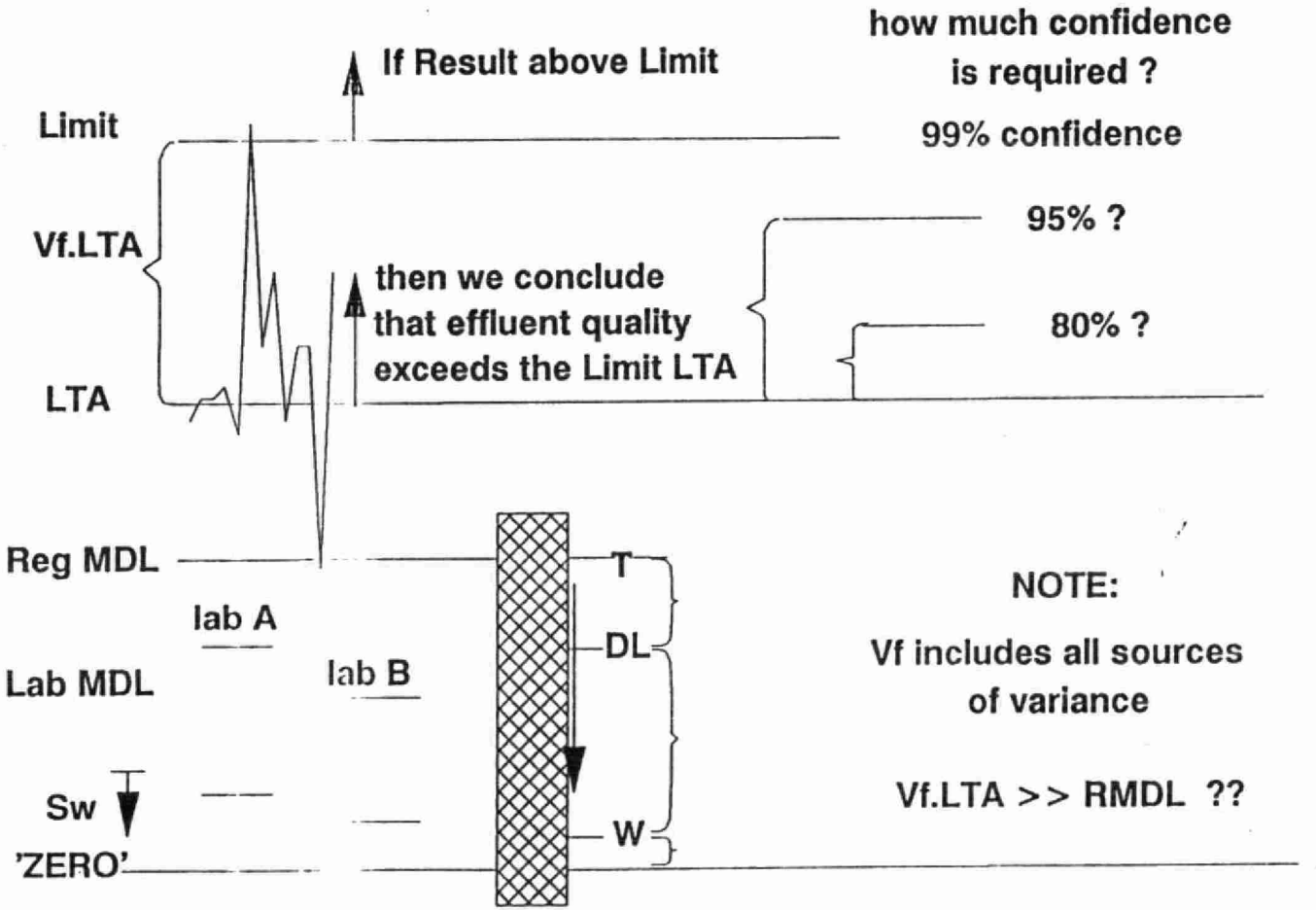
LOW-LEVEL DATA ISSUES: MISA



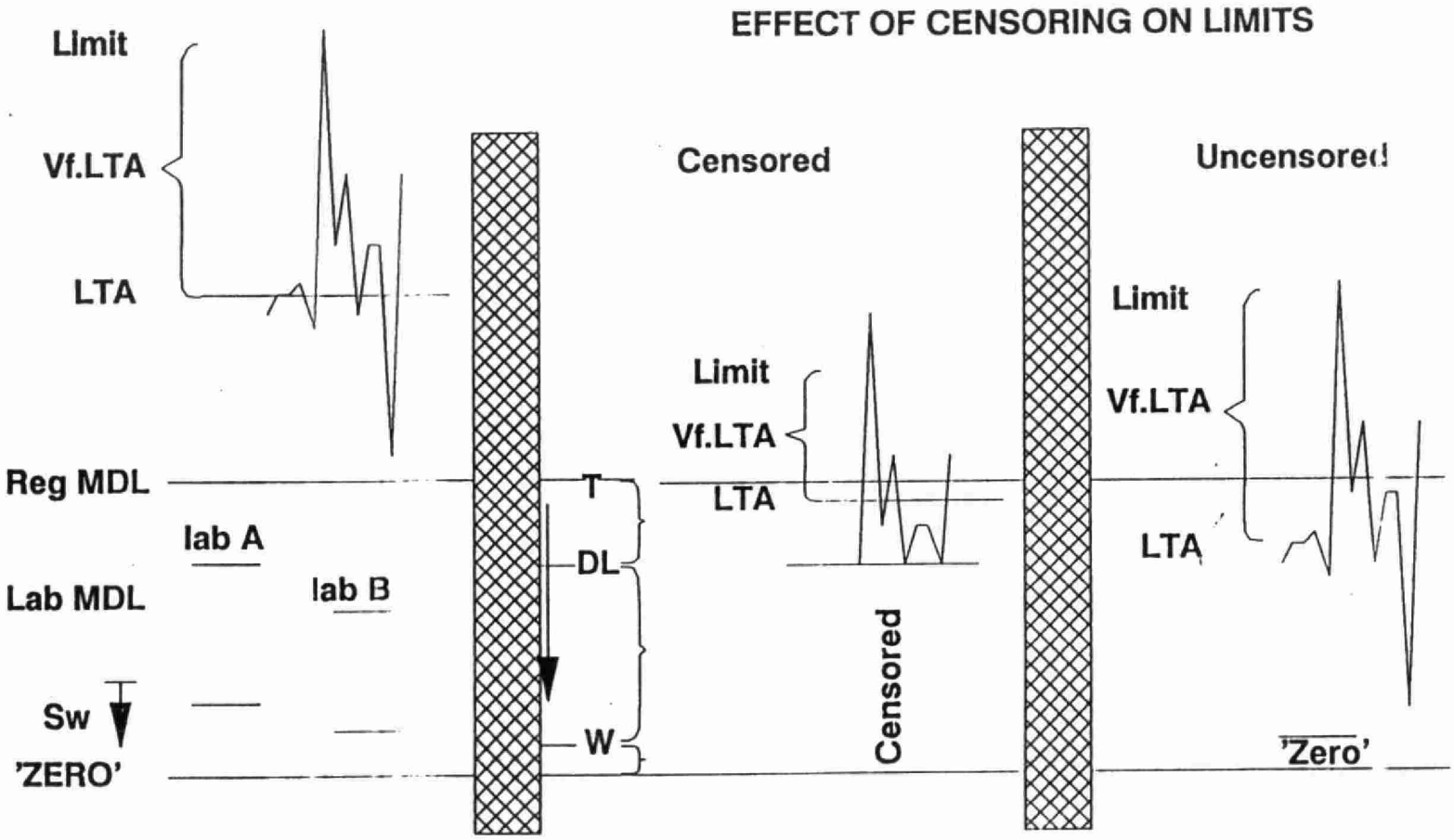
LOW-LEVEL DATA ISSUES: MISA



LOW-LEVEL DATA ISSUES: MISA



LOW-LEVEL DATA ISSUES: MISA



LOW-LEVEL DATA ISSUES: MISA

USE OF DATA

1. < Results = Unable to Measure
   Amount present unknown
2. <T Results = reported measurement below RMDL
3. <DL Results = reported measurement below Lab MDL
4. <W Results = measurement was below RMDL/10
5. Compliance decided on loadings > limit
6. Virtual Elimination versus Zero Discharge

LOW-LEVEL DATA ISSUES: MISA

SUMMARY

1. Loading Estimates should carry over Lab Remark Codes
2. For Effluent Compliance purposes Data < RMDL is not used
3. For Effluent Assessment purposes Data < RMDL is used

   Loadings based on <W Data interpreted as 'Zero'

   Loadings based on <DL, <T Data used as Calculated

   Loadings should not be calculated from < Data

4. Final decision not yet made on reporting <DL data
5. Replicate Measurements if Near Effluent Limit ?

C A E A L

1. Why formed?
2. By Whom?
3. What are its objectives, mission?
4. What is its structure and what are its programs to meet the objectives?
5. Where are we now?

THE CANADIAN ASSOCIATION FOR
ENVIRONMENTAL ANALYTICAL LABORATORIES

1. *Why was CAEAL formed?*

- concerns had been raised about data being produced by analytical Laboratories in the environmental field

- this problem had been compounded by government policies

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ENVIRONMENTAL ANALYTICAL LABORATORIES

ACLAE

CAEAL

- data quality problems were noted by various agencies during contracting out initiatives
- increase in the number of private labs each with its own QA/QC procedures or none at all
- fly-by-night labs/low bids
- IJC wrote: "*Millions of dollars are being wasted*"

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ENVIRONMENTAL ANALYTICAL LABORATORIES

ACLAE
CAEAL

WHY SUCH CONCERN ABOUT POOR DATA?

The data produced by laboratories is the starting point for the development of legislation, government policies and regulatory measures designed to protect the environment. Poor data can result in costly mistakes and affect our decisions on protecting the environment.

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CAEAL

2. BY WHOM?

In 1988, a group with representation from industry, government and universities was requested to consider the value of forming an Association through which a national QA/QC program could be developed for the self-regulation of laboratory services.

THE CANADIAN ASSOCIATION FOR
ENVIRONMENTAL ANALYTICAL LABORATORIES

ACLAE

CAEAL

- For industry, the lack of a national QA/QC program was seen as a strong negative influence on competitiveness in the industry sector (national and international)
- Governments were all for it, since they would be the main benefactors of such a program

- Governments would not need to "police" the industry as some had suggested should be done

3. MISSION

CAEAL was incorporated in June 1989 with the following mission:

to improve the quality of laboratory data, in order to develop effective policies and regulations for legislators and decision makers to protect Canada's environment.

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ENVIRONMENTAL ANALYTICAL LABORATORIES

ACLAE

CAEAL

OBJECTIVES:

- To improve laboratory analytical quality, through the provision of a national laboratory certification/accreditation program
- To provide a national forum for communication and dialogue among laboratories

- To help the industry upgrade its product and competitiveness.

1. CAEAL OPERATING STRUCTURE

a) STRUCTURE

The management team consists of a Board of Directors responsible to the membership. The board is composed of eight voting members and meets about three times yearly.

A secretariat provides support to the Board and each of the program committees.

An Advisory Panel recommends to the Board re: certification or suspension of certification.

Each program area is under the direction of a program committee appointed by the Board.

The day to day operation is handled by a Program Manager and an Office Assistant.

b) FINANCIAL CONSIDERATIONS

CAEAL is funded by membership fees and fees charged to laboratories seeking certification. CAEAL has received financial support and continues to receive in kind support from governments and private industry. CAEAL operates on a cost-recovery basis.

c) CANADIAN COUNCIL OF MINISTERS OF THE ENVIRONMENT (CCME)

The CCME has given strong support to CAEAL. It has instructed their members right across Canada to use CAEAL Accredited laboratories. The CCME encourages laboratories in each province and territory to become members of CAEAL.

d) PROGRAMS - QA/QC

CAEAL has, as its principal objective, the promotion and maintenance of a strong environmental analytical service within Canada. A vehicle for achieving the objective is provided by the Laboratory Certification/Accreditation program offered by CAEAL.

PHASE 1: *CERTIFICATION*

Certification is the formal recognition by the Association of the proficiency of an environmental analytical laboratory to carry out specific tests. Under this program, participating laboratories are sent test samples at six month intervals for analyses.

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The certification granted relates solely to the specific tests for which a laboratory seeks certification and for which certification is available.

PHASE 2: *ACCREDITATION*

- In 1992, CAEAL will be expanding its program to include not only the provision of test samples, but also site visits to observe the actual operation of laboratories

- Protocols are being developed to conduct periodic site visits by qualified assessors

- Laboratories, which successfully meet the national standards associated with site inspections and analyses of test samples, will be granted accreditation which will replace the certification offered at this time.

5. WHERE ARE WE NOW?

CAEAL initiated the Certification program early in 1991 and granted Certification to 40 laboratories in Canada. This number is increasing as the program develops and more tests are added to the list of those offered for certification. 64% of participating laboratories are from industry, 19% federal and 17% provincial laboratories.

All provinces were represented, except PEI.

CAEAL has published a Directory of Certified Laboratories.

- The first of samples were shipped in Feb. 1991 and the maintenance program calls for testing every 6 months

- For our last study in September 1991, we had 34% increase in the number of participating laboratories and there are now 55 labs in the program

- Samples with organic constituents are being developed for distribution in 1992

OTHER PROGRAMS

- EDUCATION
- STANDARD REFERENCE MATERIALS
- RESEARCH AND DEVELOPMENT
- INTERACTIVE
- RESOURCE DEVELOPMENT

BENEFITS TO MEMBERS

- Provide a national standard of competence through the certification program
- Provide national recognition for successful laboratories through the publication of an annual "Directory of Certified Laboratories"
- Enhance product quality and reputation of member laboratories

THE CANADIAN ASSOCIATION FOR
ENVIRONMENTAL ANALYTICAL LABORATORIES

ACLAE
CAEAL

- Assist industry to compete in international markets
- Provide services to members in the areas of QA/QC, education and training, standard reference materials, research and development and communication
- At a minimum, the establishment of uniform performance standards will provide analyses with a fixed bench-mark against which to measure their performance on a continuous basis

The Canadian Association for Environmental Analytical Laboratories

WE'VE MOVED!

From: Suite 404C - 1 Nicholas
Ottawa, Ontario
K1N 7B7

To our new office

Suite 532 - 1 Nicholas Street
Ottawa, Ontario
K1N 7B7

Phone: (613) 562-2200
Fax: (613) 562-2203

SECTION FIVE

QUALITY MANAGEMENT PRESENTATIONS

CONTROL CHARTING ISSUES - D. KING, MOE

EVALUATION OF INTER-LAB STUDIES - S. SELLIAH, MOE

MISA WORKSHOP 1992

CONTROL CHARTING ISSUES

Don King

DISCUSSION

This presentation shows diagrammatically the rationale for control charting, namely the need to take timely action to limit the relative magnitude of between-day versus within day estimates of precision, to detect unusual events (outliers) and to forestall bias as revealed by drift or discontinuities. It demonstrates the relationships among Shewhart, Youden, and King's approaches. It reviews the process for setting control limits based on within batch repeatability (Sw) as estimated by the mathematical standard deviation.

Fig.1

Performance is often discussed in terms of the sharpshooter's bulls-eye. Ability to hit the centre reflects accuracy. Spread across the target indicates precision. Since no one is absolutely accurate, accuracy is a characteristic of average performance. If the average deviates significantly from the expected value, the performance is biased. Significance is based on the estimate of repeatability (Sw) and the number of replicates included in determining the average. The two small circles (A, B) represent apparent or actual changes in repeatability from day-to-day for a given analyst, or between analysts, or between methods for a given analyst.

When replicate measurements are performed over several batches/runs, or by several analysts/labs, the precision is observed to degrade. This reproducibility is attributed to the day-to-day variation in restandardizing the measurement system within a single lab (middle large circle), or to the variability of standards among labs (bottom large circle).

In the middle large circle, the small circles represent the variable estimates of repeatability. These are centred at the actual bias point for which a standardization adjustment may be required. The line joins the individual estimates used to restandardize. The large circle then represents the average reproducibility achieved. One aspect of CONTROL is to reduce the size of this circle by controlling daily restandardization decisions.

The lower large circle represents reproducibility among laboratories. In addition to the previous effect, each lab has a different source of calibration material which must be diluted and prepared for use as a daily standard. This induces a source of bias which degrades data comparability among labs, and which also must be controlled.

Fig.2 Youden 2-Sample Plot

Youden's two-sample procedure, developed in the early '50s for interlaboratory comparison purposes, provides evidence for the bias among labs. The result for sample A is plotted versus the result for sample B from the same analyst. Typically one sees a pattern of points from lower left to upper right. This evidence of bias is generally attributed to the differences among standards in different labs, or to changes in method recovery. Results in the other two quadrants are considered very imprecise or erratic. When the two samples are similar in concentration these points fall along a 45 degree axis. The perpendicular distance from a point to the 45 degree line is an estimate of that lab's repeatability. The distance along the 45 degree from the perpendicular to the expected/target point represents the lab's bias. The average

perpendicular distance can be used to determine average repeatability for the participating analysts as indicated by the solid circle surrounding the target point. When the median value for each sample deviates significantly from the target value, the data can be used to set a consensus point among the labs. An analyst deemed as 'biased high' relative to the consensus point may be deemed 'acceptably accurate' relative to the target point.

Fig.3 Shewhart Control Charts

In the late '20s, Shewhart introduced the time sequence control chart in which he made replicate measurements for a particular characteristic each day, and plotted the range (R) and average (X-bar) over a period of time. In the lab this requires establishing a control sample which is analysed twice each day. The top diagram shows the actual replicate measurements. Many would ignore the apparent trend over time. But Shewhart's plot of the average shows the trend quite clearly.

Performance limits are determined by using the average range (excluding outliers e.g. > 3 R_{avg}) to set upper and lower control limits (UCL, LCL) and warning limits (UWL, LWL) for the average. Note that the average range can be used to estimate repeatability (Sw = 0.886*R_{avg}). The control limit for the average is then 3*R_{avg}*0.886/1.414 or 1.88*R_{avg}. The root 2 factor reflects increased confidence in the average of two values compared to a single value.

Fig.4 Systematic Errors

In the early '70s King introduced the use of two control samples for controlling daily standardization in the lab. One is at the upper end of the operating range: the other is at the lower end. They should be close enough together that the repeatability at the two concentration levels is not significantly different. Such time-sequence control data can be plotted using the Youden approach.

When the two samples differ by more than a factor of two (e.g sample A = 25 and sample B = 50 mg/L) King observed that there are two '45 degree' lines. One represents a bias which is equal for each sample in absolute terms. (Both sample results are low by 5 mg/L for a given analyst.) This type of bias results from an error in setting the baseline, background, or blank correction factor. It appears at 45 degrees if the X-Y scales are set so that 1 mg/L is a constant distance, leading to a rectangular presentation.

The other line represents a bias which is constant in relative terms. (Both sample results are low by 5% for a given analyst.) This type of bias represents an error in preparing or using standards, (lack of calibration control) or certain types of method failure. This line becomes a 45 degree axis when the X-Y scales are set so that 1% is the same distance on both, leading to a square diagram.

By joining the points in time sequence the systematic drift from day to day is accentuated by the many line segments which more or less parallel the bias line from lower left to upper right.

Fig.5 Two Sample Control

The data used to plot figure 4 is shown here as two time sequence plots for sample A and sample B. Also shown are the Sum plot (A+B) and the difference plot (A-B). In this case the difference represents both the repeatability of the test and the slope control status, and the effect of systematic error is essentially cancelled on average. (Note that these are signed differences.) Assuming that the repeatabilities at level A and level B are sufficiently similar, then the standard deviation of the differences S_D is approximately 1.414 larger than Sw as noted by Youden.

Control and warning limits for the A-B plot are set at 3x and 2x S_D. Outliers represent a sudden change in slope or erratic analysis. The average difference indicates stability of slope and an indication of bias relative to expected.

The A+B plot includes a double dose of bias. But when the A-B plot is stable, any large variation in the A+B plot must be attributed to errors in setting baseline or method blank corrections. On the basis that the ratio of two variances is significant when it exceeds a factor of 2, it was decided to allow a factor of 1.33 times the repeatability as an acceptable level of between-day reproducibility. Therefore the control limit for the sums (A+B) is set at $3*1.33*S_D$ or $4*S_D$. The warning limit remains at $2*S_D$.

Fig.6 Single Sample Control

Shewhart Control requires at least duplicate analysis of a single control sample. But frequently the analyst has only a single analysis per day. Such data can be plotted in time sequence, and the average and standard deviation (S) can be determined. But such a plot is NOT a control chart in as much as the calculated S is used to set limits. This is demonstrated by the top two diagrams. They both contain the same data but in different sequence, therefore the average and standard deviation are identical. But there is obvious drift in the left hand data set, and there may be a discontinuity shift in the right hand data.

An estimate of repeatability (Sw) is always required to set proper control limits. This may come from other within-run replication data. But it can be estimated from the existing data by examining adjacent pairs (S_{adj}). The lower diagrams then are similar to Shewhart's range chart in calculating an average range of adjacent pairs, and using it to set a control limit for the single sample time sequence chart, namely $3*0.886*R_{avg}$. One would not add the factor 1.33 used in the King two-sample approach because the range of adjacent pairs is probably an overestimate of repeatability. Note that the lower of the two estimates (S or S_{adj}) is used to set limits.

SUMMARY

Bias is readily detected when sufficient replicate measurements are made. The ratio of reproducibility over repeatability indicates the level of control over determinate sources of error/bias. Control is directed at reducing this ratio on average, forestalling significant drift over time, preventing discontinuities, and reacting to outliers as possible evidence for inadequate system operation. Good control is based on two principles:

a) leave well enough alone (don't fix it if the data is within control limits),

b) don't fix it if you don't know what went wrong (have a remedial action plan).

REPEATABILITY

within-day

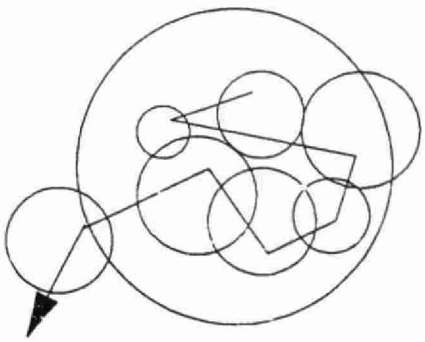
Sw

REPRODUCIBILITY

within-lab
between-day

S

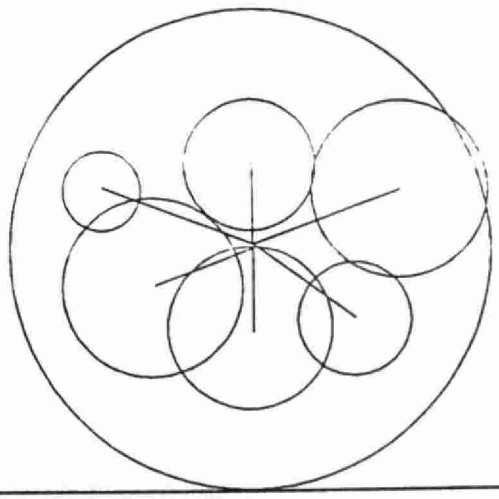
standardization
induced drift ?



REPRODUCIBILITY

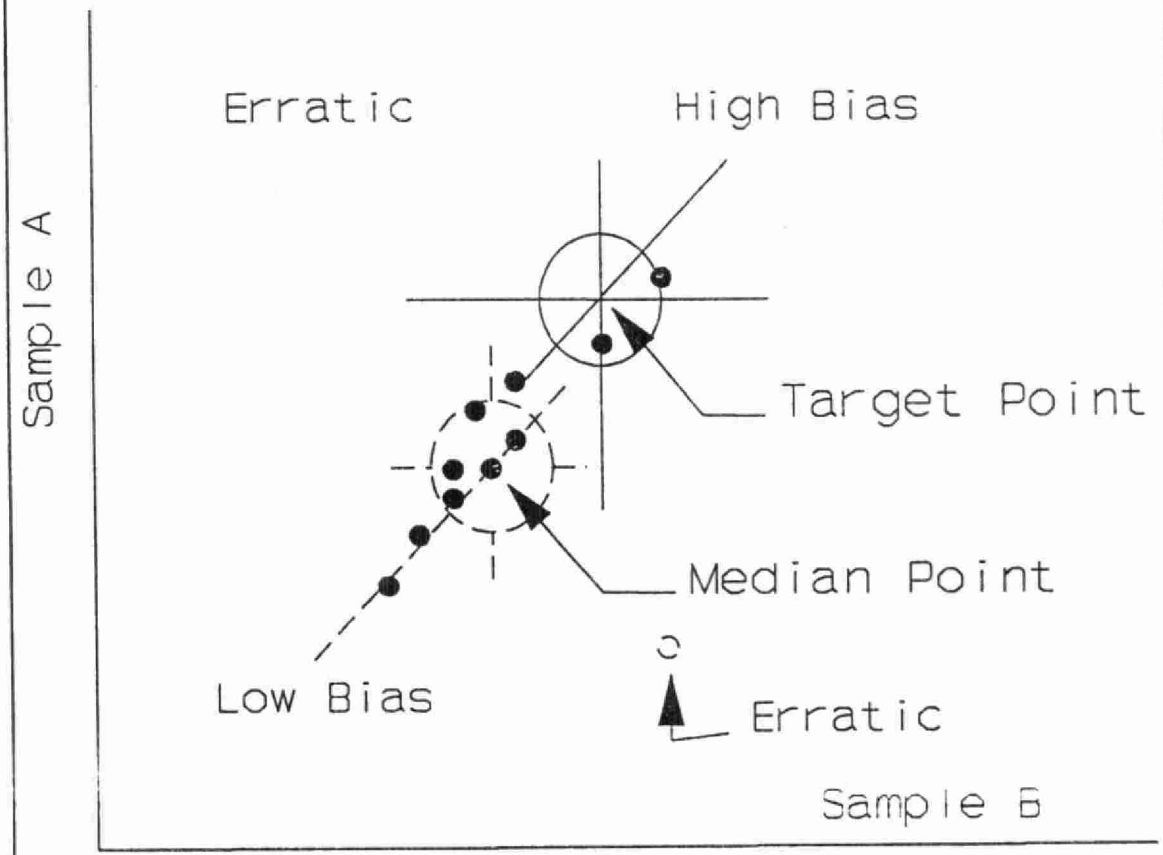
between-lab

bias in
standards ?

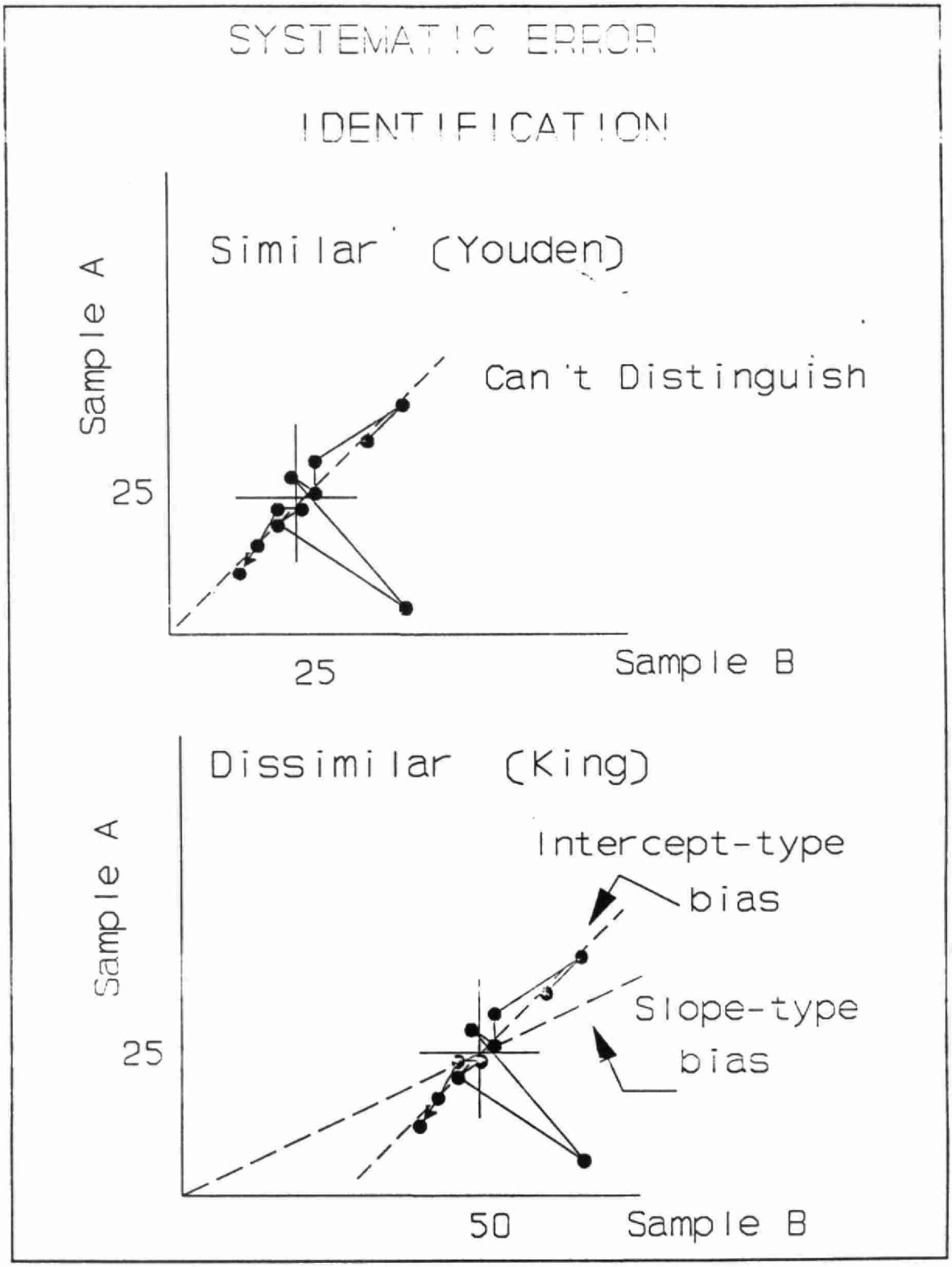


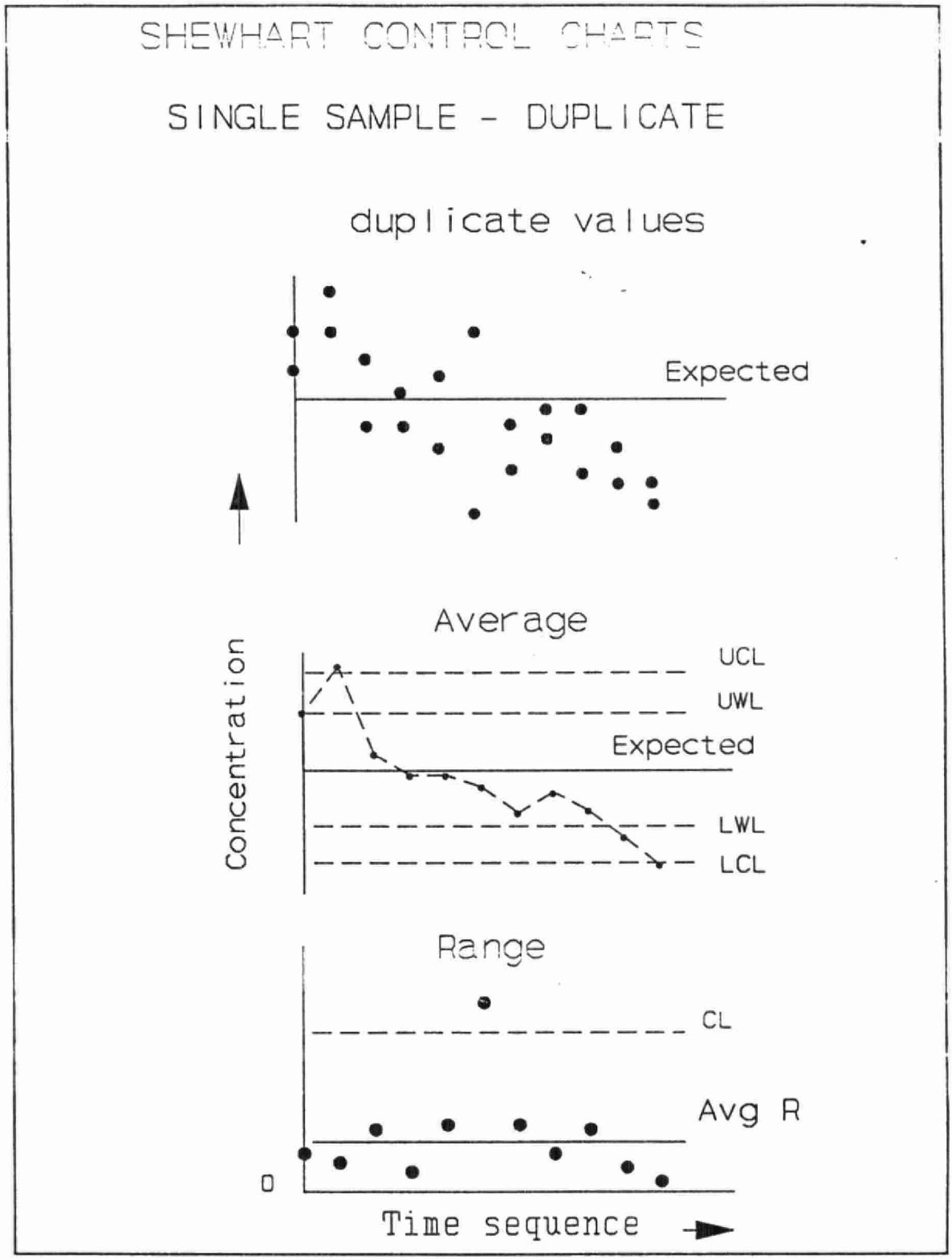
YOUDEN TWO SAMPLE PLOT

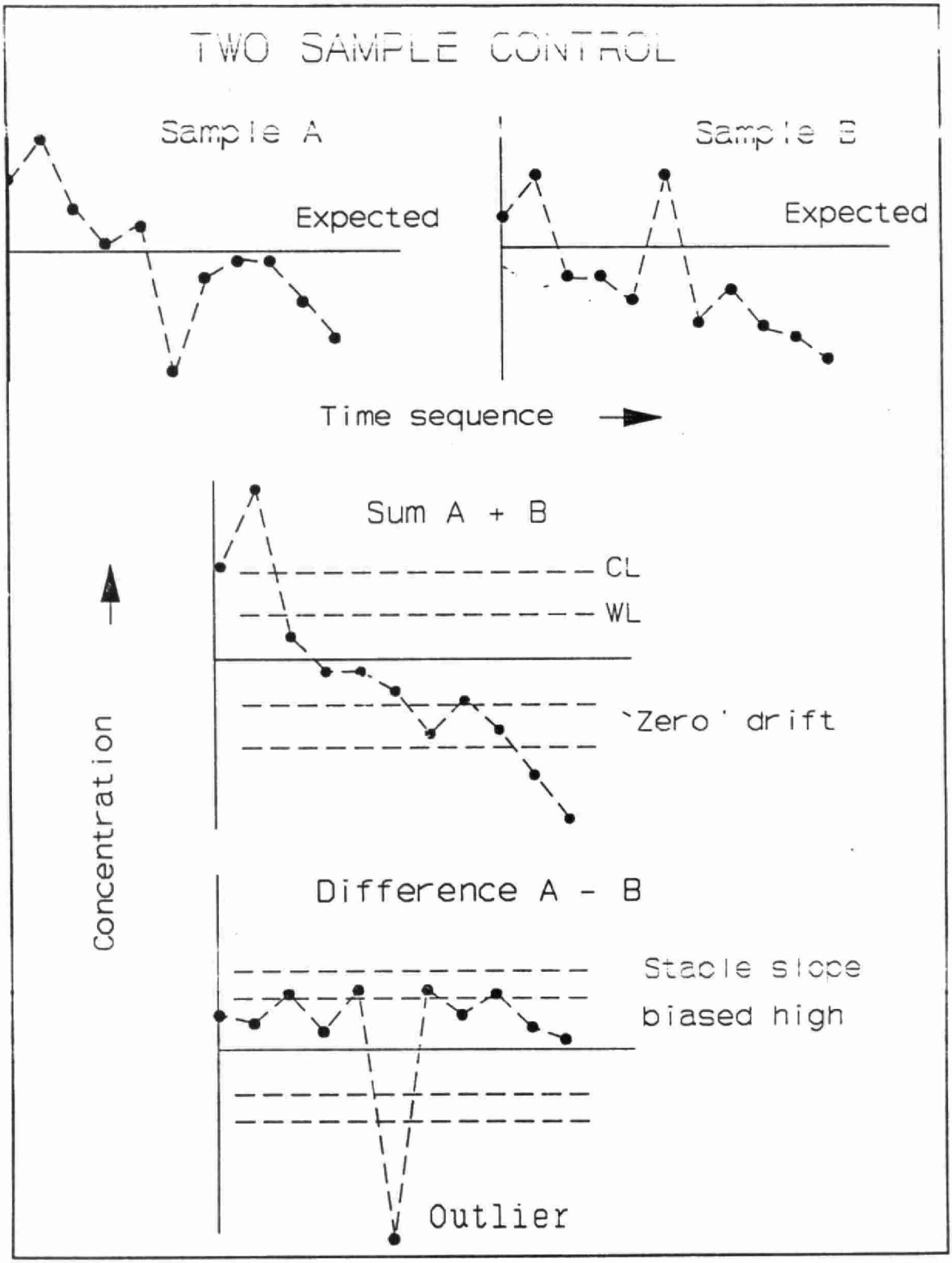
INTER-LABORATORY STUDY



circle = warning limit
for repeatability


SYSTEMATIC ERROR
IDENTIFICATION
Similar (Youden)
Can't Distinguish
Sample A
25
25
Sample B
Dissimilar (King)
Intercept-type
bias
Slope-type
bias
Sample A
25
50
Sample B

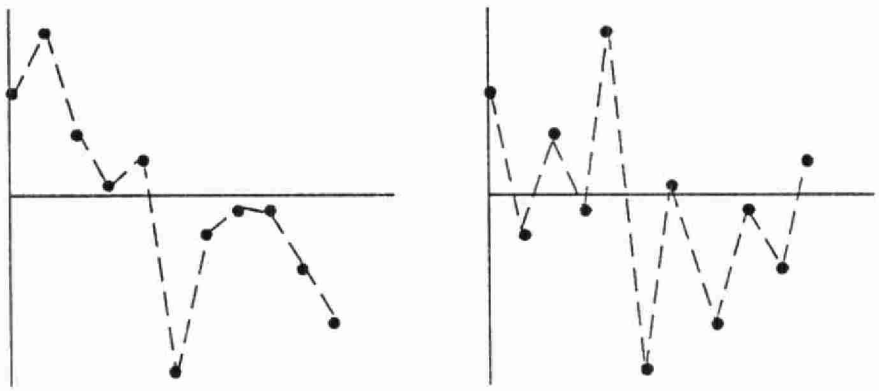

SHEWHART CONTROL CHARTS
SINGLE SAMPLE - DUPLICATE
duplicate values
Expected
Average
Concentration
UCL
UWL
Expected
LWL
LCL
Range
CL
Avg R
0
Time sequence


TWO SAMPLE CONTROL
Sample A
Expected
Sample B
Expected
Time sequence
Sum A + B
CL
WL
'Zero' drift
Concentration
Difference A - B
Stacle slope
biased high
Outlier

SINGLE SAMPLE CONTROL

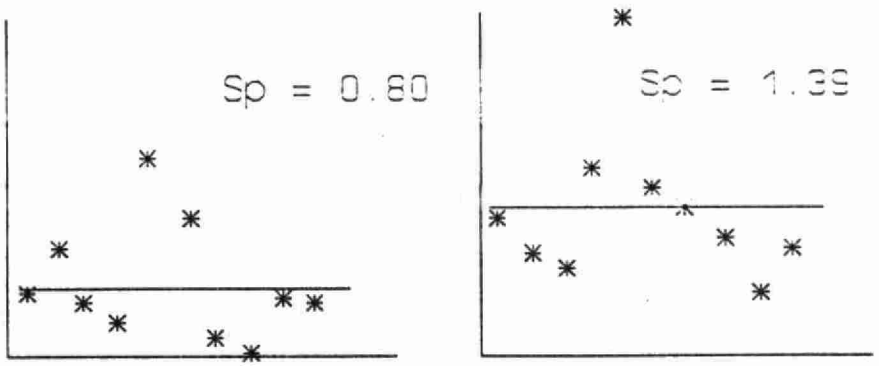
Same data - Different Order

average = 82.9 S = 1.19



apparent trend random ?

Difference between adjacent pairs



Time sequence ► Time sequence ►

A RAPID AUTOMATIC GRAPHICAL PROCEDURE FOR THE EVALUATION OF INTERLABORATORY STUDIES

SATHI S. SELLIAH

Historical background:

SLIDE 1

In the 50's Youden proposed a scheme where each participant analyses two similar samples. They are similar in concentrations. When he received the results he plotted them on a X_Y chart. Each point on this graph represents a pair of results from a participant. He divided the graph into four quadrants by two lines going through the respective medians. If there are only random errors one would expect the points in the graph to be equally distributed among the four quadrants.

SLIDE 2

However in actual practice he found that most of labs were in the lower left or upper right quadrants and that they tend to lie along the 45 degree diagonal line. If a laboratory reported lower than expected for one sample it was very likely that it will report lower than expected for the other. Likewise if a laboratory reported higher than expected for one sample it is very likely that it will report higher than expected for the other. The systematic errors among the laboratories were very evident.

An acceptance circle based on some factor times the standard deviation is drawn. Laboratories inside the circle are deemed satisfactory. Laboratories outside the circle and close to the 45 degree line are biased high or biased low. Laboratories outside the circle and lie in the upper left and lower right quadrants are erratic.

SLIDE 3

Don King recognized that the Youden plot can be used to isolate two types of systematic errors: those that are dependant on the concentration (slope errors) and those that are not dependant on the concentration (intercept errors). Slope errors can be attributed to calibration errors (inaccurate standards or inappropriate procedure) or recovery problems. Intercept errors can be attributed problems associated with blank's, background or baseline corrections.

In order to monitor these two errors on a day to day basis a two sample control procedure was introduced to the MOE laboratories. Two controls A & B which are different in concentrations are analyzed just after the calibration standards, each time a new run is set-up. The results of these standards are plotted as shown and the points are joined sequentially . The emerging pattern of the AB plot reveals the type of systematic errors if there are any.

In these graphs there are two diagonal lines. The line that goes through the origin represents identifies slope biases and the 45 degree line identifies the intercept biases. If there are no biases, the AB pattern will take a circular shape.

SLIDE 4

The previous two approaches have been combined to evaluate inter-lab data. Here too, each laboratory is required to analyze two samples, but the samples are of different concentrations. This primarily enables us to distinguish between the two types of systematic errors identified earlier.

While inter-lab reproducibility S_r tends to be concentration dependant, the repeatability S_w may not be concentration dependant. The dependence of repeatability on concentration is assessed by determining the reproducibility for each sample after excluding the outliers and determining the ratio of the S_r. '*F* statistic'is used to assess if the difference between the two S_r's are different. If S_r of the two are not significantly different it is concluded that the S_w is not concentration dependant.

When repeatability is independent of the concentration the raw data can be used as such (that is in concentration units or absolute scale) to draw the graphs and define the acceptable performance criteria. The

resulting diagram takes a rectangular shape. When repeatability is concentration dependant, then the raw data must be transformed into a more comparable scale before acceptable performance criteria could be determined. In such cases the results will be expressed as percentages of target (or median) resulting in a square graph. This ensures that the acceptable repeatability zone remains essentially circular in shape.

In both cases you can identify the two diagonal lines representing the two types of errors.

SLIDE 5

The next step is to calculate the average within-laboratory repeatability S_w. This is done by calculating the perpendicular distances from each point to the appropriate diagonal line and averaging these PDs. A screening process is involved at this step to eliminate highly imprecise or erratic data. Actual steps involved are summarized in slides 7 & 8.

SLIDE 6

The S_w is used to define the warning and control limits. The inner circle represents the limits for repeatability.

We recognize that additional tolerance is required to allow for the variability in preparing and using calibration standards on a day to day or among labs basis. This is the reproducibility component.

A factor 1.5 was for the ratio of reproducibility to repeatability is considered an achievable goal for interlaboratory reproducibility and accordingly the area of acceptable performance is extended in the lower left and upper right quadrants have been extended by a factor of 1.5.

The evaluation of a laboratory in this procedure will depend on its position on the graph.

Laboratories that lie within this key hole shaped shaded area will be considered as acceptable performers.

Laboratories that have blank problems will lie close to the intercept bias line.

Laboratories that have calibration problems will lie along the concentration bias line.

Laboratories that lie close to any of the median lines have one of their results correct and the other in error. They are clearly out of control,

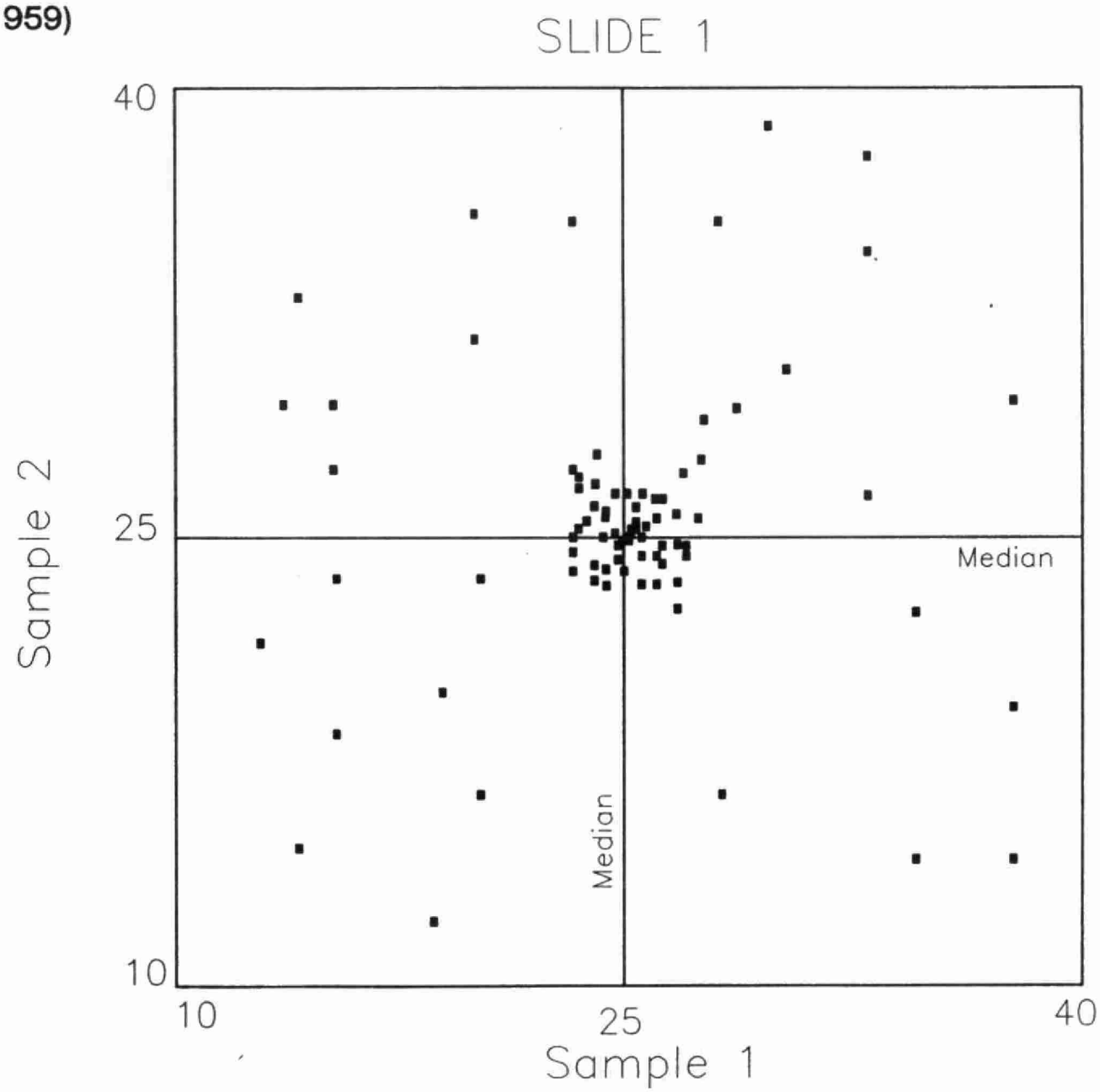
The laboratories in the upper left and lower right are highly imprecise or erratic.

SLIDE 9

A main feature of this method is that it is designed to prompt immediate remedial action when problems are identified.

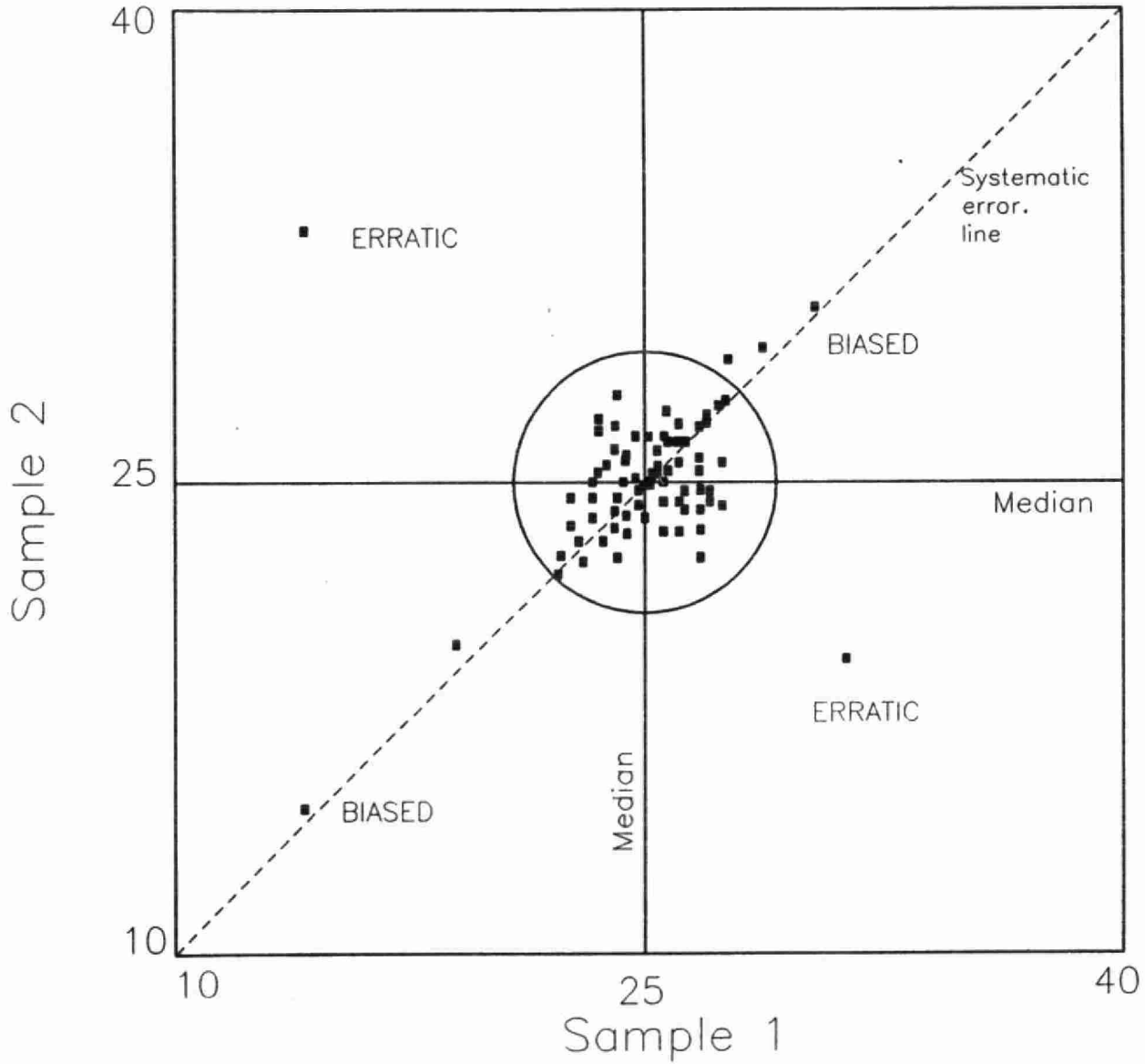
All the steps I have described can be automated.

YOUDEN (1959)



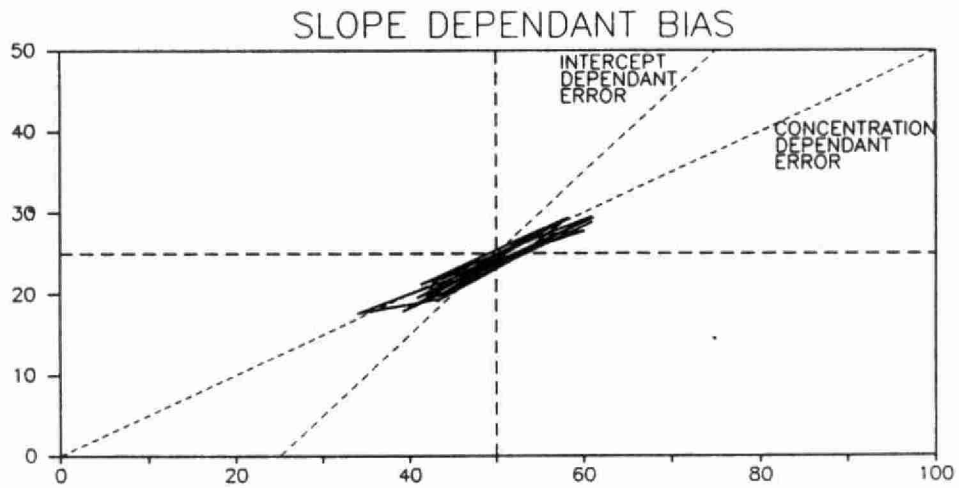
YOUDEN (1959)

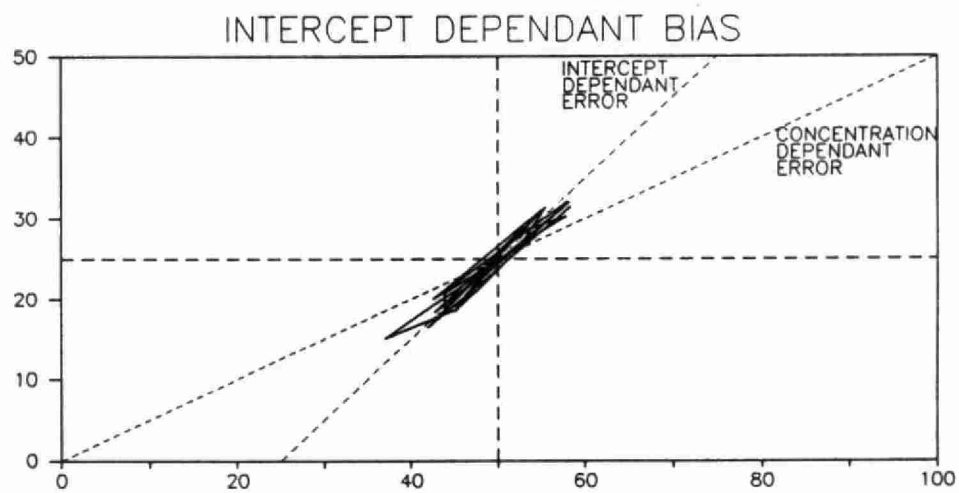
SLIDE 2

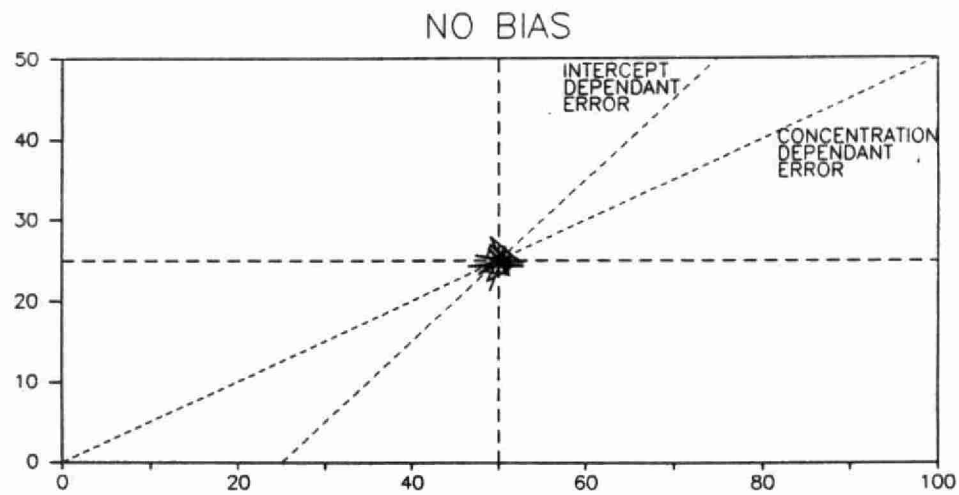


SLIDE 3

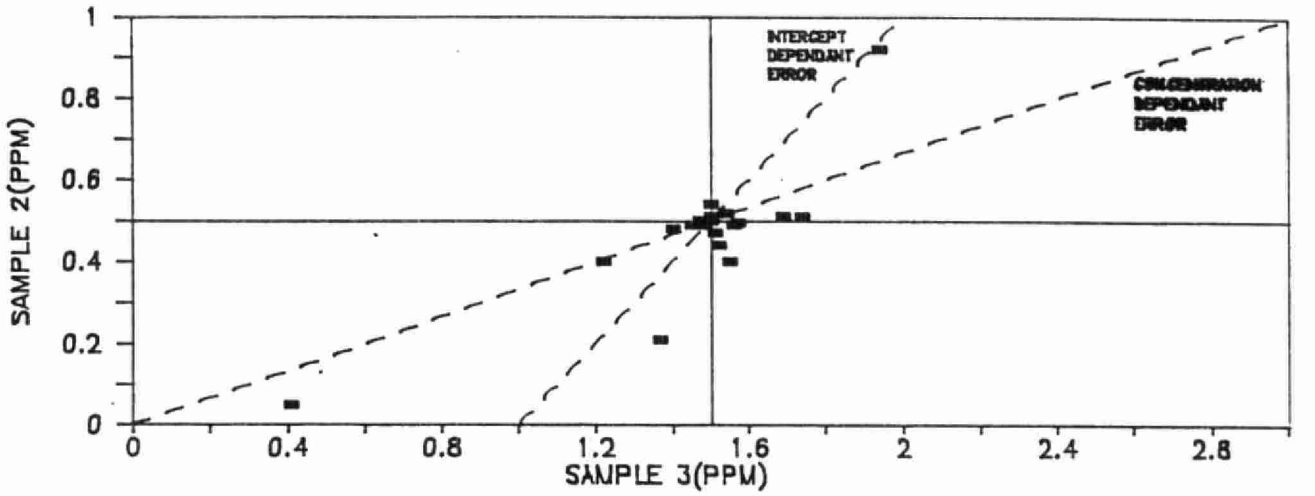
KING (1970)



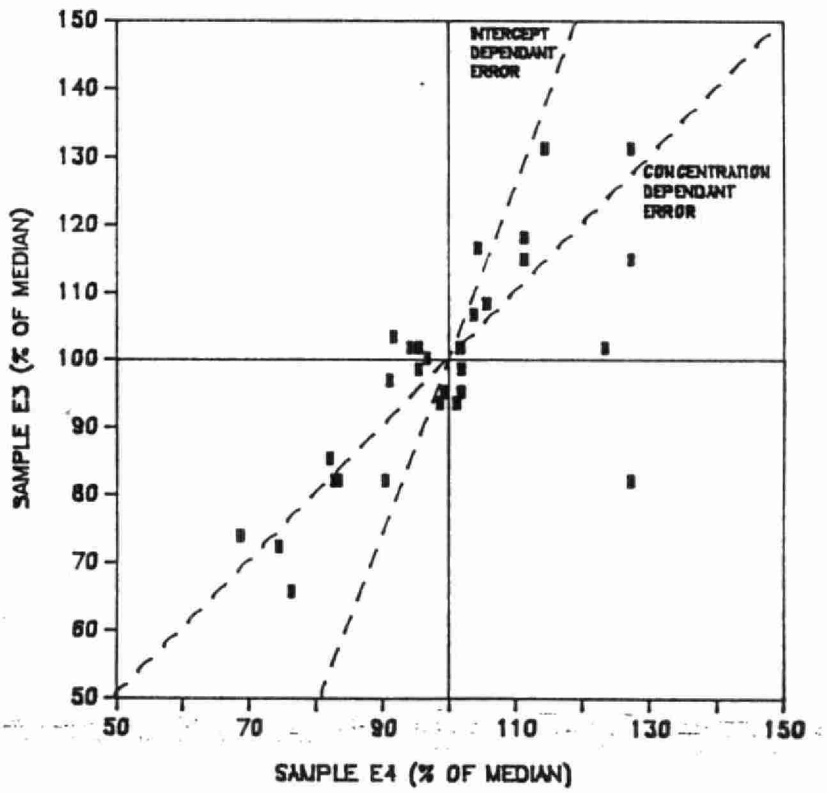




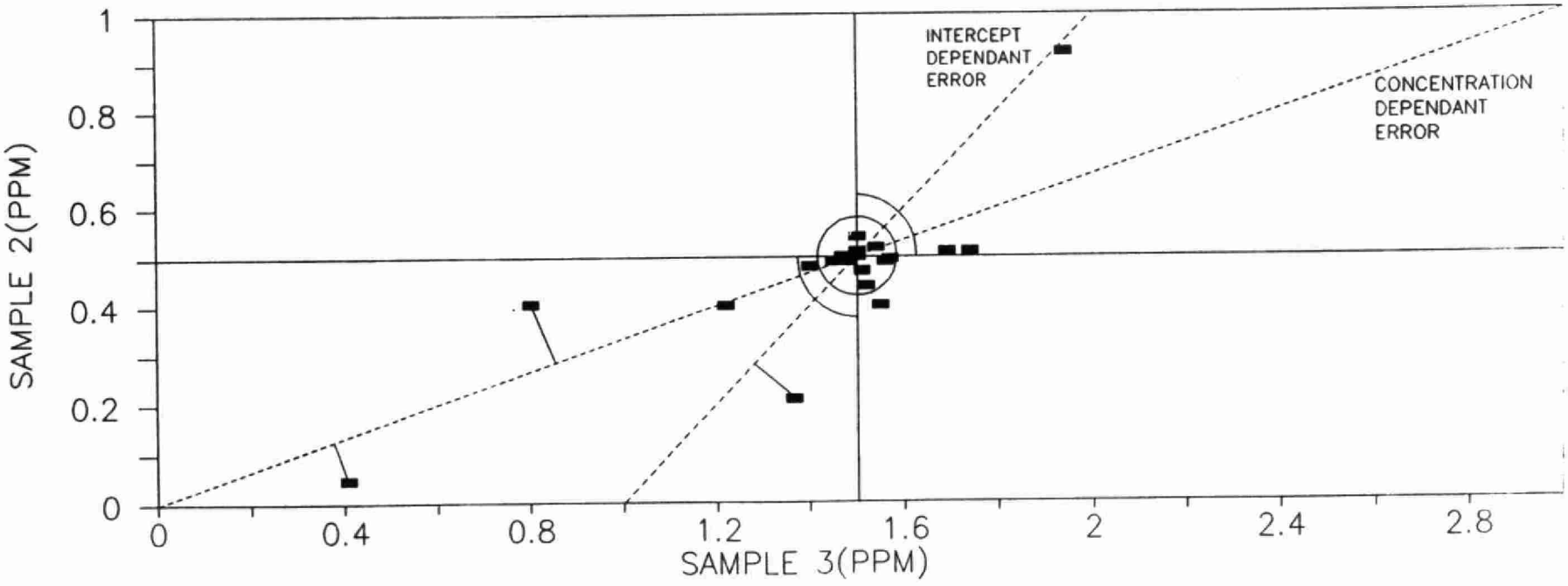
ABSOLUTE SCALE



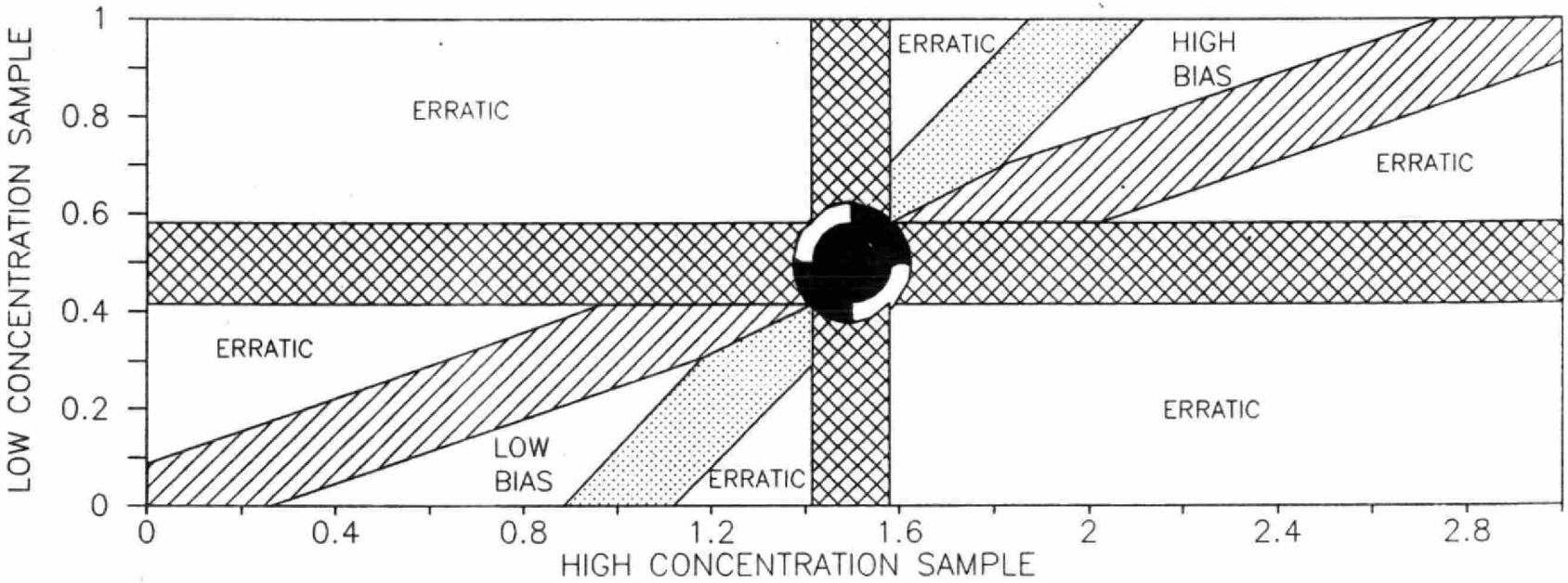
RELATIVE SCALE



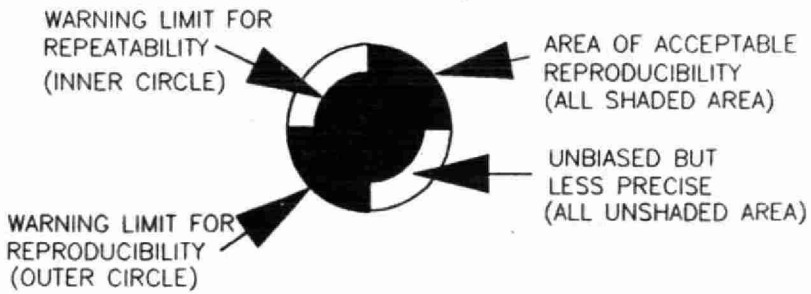
SLIDE 5

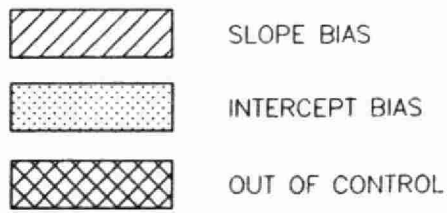

INTERCEPT
DEPENDANT
ERROR
CONCENTRATION
DEPENDANT
ERROR
SAMPLE 2(PPM)
SAMPLE 3(PPM)
0
0.2
0.4
0.6
0.8
1
0
0.4
0.8
1.2
1.6
2
2.4
2.8

SLIDE 6



KEY TO SHADED AREAS





Step-wise Summary of The Evaluation Procedure

1. Sample preparation and distribution

Split two samples of different concentrations among a number of laboratories for analysis/measurement using their current methodology.

2. Enter data on LOTUS 123® spreadsheet.

Calculate median (L_m,H_m), means and standard deviations for each sample.
Tabulate data and return to laboratory analyst for verification.
Correct database if transcriptional errors were reported.

3. Evaluate high sample data:

i) reject all results which differ from the median (H_m) by more than 10%
ii) calculate median(H), mean and standard deviation (S_h)
iii) re-include data if within 3 times S_h
iv) reiterate ii) and iii) until no further data is included
v) calculate relative standard deviation of the final selected data (CV_h)

4. Evaluate low sample data:

i) use 3 x CV_h x median(L_m) to exclude possible outliers
ii) calculate median(L), mean and standard deviation (S_l)
iii) reinclude data if within 3 times S_l)
iv) reiterate ii) and iii) until no further data is included.

SLIDE 8

5. Determine paired sample performance criteria:

i) examine the ratio of S_h/S_l and if:
<2 use data as reported in concentration units
otherwise convert to % recovery based on expected value if known (otherwise use median values (H,L))
ii) prepare paired sample scatter diagrams of all data
iii) calculate perpendicular distances from each point to the two 45 degree lines (slope and intercept error lines) and select the lesser of the two perpendicular distances (PD)
iv) determine the median PD
v) determine the average of all PD values less than 2.5 times the median and use this average to estimate the average repeatability S_w.
vi) set warning limits for repeatability = 2 times S_w
vii) set control limits for repeatability = 3 times S_w
viii) set warning limits for possible bias = 3 times S_w
ix) set control limits for possible bias = 4.5 times S_w

6. Code performance based on location of points on the diagram using LOTUS 123® program:

i) in upper left or lower right quadrant (erratic)
ii) in lower left or upper right quadrant (biased low or high)
iii) on horizontal or vertical axis (out of control)
iv) on diagonal line through origin (slope or standard problems)
v) on diagonal line not through origin (intercept or blank problems)
vi) prepare summary table of performance assessment

SLIDE 9
KEY FEATURES

ACTION FOCUSED PROTOCOL

- **A**utomated evaluation protocol

  The procedure is easily amenable to computation

- **C**ost per participant is minimum

  Each laboratory is required to analyze only two samples

- **T**ime required for evaluation is minimum

- **I**dentifies and classifies the types of errors found among laboratories

  Facilitates remedial action

- **O**utcomes presented graphically in a convincing form

- **N**o statistical background required to follow reasoning